LIQUID-LIQUID EXTRACTION IN A PULSED PERFORATED-PLATE COLUMN

A THESIS

Presented to

the Faculty of the Graduate Division

by

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in Partial Fulfillment

of the Requirements for the Degree

Doctor of Philosophy

in the School of Chemical Engineering

Georgia Institute of Technology

August 1952

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PULSED PERFORATED-PLATE COLUMN

Approved:

Date Approved by Chairman: Aug. 26, 1952

ACKNOWLEDGEMENTS

For his aid and guidance in the prosecution of this work, the author wishes to express his sincere appreciation to Professor Wm. M. Newton. The author is indebted to Professor J. M. DallaValle, Professor W.T.Ziegler, and Professor K.M.Murphy for many suggestions on the writing of this thesis.

The author should also like to thank Messrs. R.A.Dysart, G.A.Kloepfer, M.De Laguardia, B.M.Martin, J.B.Powers, M.Raynes, G.T.Rosser, R.A.Samoden, R.H.Slaughter, and R.L.Tagna for their help in building the equipment and obtaining the data.

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SUMMARY

Liquid extractions of toluene-benzoic acid solutions by means of water were studied under varying flow rates of both solvents in a 2-in. perforated-plate column to which pulsations of different frequency and amplitude were applied. Studies on reversal of phase of dispersion by making toluene and water as the discontinuous phase were made. With fixed flow rates of both solvents, the rate of extraction increased much more rapidly when the pulsation through the perforations became turbulent. The extraction coefficients were not affected by the individual values of frequency or amplitude of pulsations, and by the product of the two. Data on toluene hold-up and individual plate efficiencies are included. The results are correlated by the equations:

$$(HTU)_{ow}(Re)^{l} = \frac{HV_w}{\phi_T(V_T)^n} + \frac{1}{\phi''_w(V_w)^{s-1}}$$

$$\frac{K_w a H}{(Re)^{l}} = \phi''_T(V_T)^n\,(V_w)^m$$

INTRODUCTION

There are several general operations available to chemical engineers for separating a liquid solution into its component parts. Fractional distillation is perhaps the operation most frequently resorted to when the components of the solution are volatile. In certain cases, however, substantially complete separation by fractional distillation can not be achieved due to the formation of an azeotropic or a constant boiling mixture. In other instances, when the solute in a solution is less volatile than the solvent, or the concentration of the solute is low, the recovery of such a solute by using fractional distillation would require the expenditure of large quantities of heat to vaporize the solvent and would make such operation uneconomical.

Crystallization is used to separate a non-volatile solute from a solution, but it is usually limited to solutions with high concentration of the solute.

Some other operations such as adsorption, evaporation, diffusion and precipitation are used for the separation of a solute from its solution, but they are also found at times inapplicable to some solutions due to physical or chemical limitations.

It frequently happens that the nature of the original solution or the costs involved in such separation will prevent the application of any one of the processes described above, and then liquid-liquid extraction is usually found to be useful.

Industrial Applications.—In petroleum refining, the initial separation of different fractions in the crude is accomplished by fractional distillation which, however, makes substantially no segregation of its components according to chemical type. Separation of these by using chemical reagents is commonly used; for example, the action of sulfuric acid on unsaturated hydrocarbons. Recently, liquid-liquid extraction has been extensively used to replace this chemical process of separation. In petroleum oils, objectionable sulfur compounds such as hydrogen sulfide, mercaptans, and others may be removed by liquid extraction.

In the manufacture of synthetic rubber, ordinary distillation can not be used to separate the relatively pure 1, 3-butadiene from other C_4-hydrocarbons, while liquid extraction has found extensive use.

Extraction processes have been used in separating the constituents of many oils, fatty acids and related substances; for example, the separation of the saturated from the unsaturated glycerides of vegetable oils by using furfural as solvent.

In the operation of by-product coke ovens, the gas evolved during coking is sprayed with water and cooled, thus depositing an aqueous solution called "gas liquor". This gas liquor contains in solution some phenols which must be removed before the solution can be discharged into the sewer, since phenols are harmful to fish and affect the taste of the water. The phenols are therefore removed from the gas liquor prior to distillation by extraction with a light oil.

There are many organic chemical manufacturing processes using liquid extraction as a step in the procedure. A few examples are cited to indicate the wide applicability of extraction process in industry. The following may be mentioned: solvent extraction of tar acids from coal tar hydrocarbons (39), toluene extraction from petroleum with water solutions (40), solvent extraction of thiodiacetic acid from water solutions (41), separation of m- and p-cresols by liquid extraction (42), and extraction of lactic acid from water solutions by amine-solvent mixture (43).

A more detailed discussion of industrial applications of liquid extraction may be found in Treybal's recent book(51).

Review of Literature and Historical Background of the Present Investigation.—Under the continuous counter-current type of extraction equipment, a large number of designs have been proposed, as evidenced by the considerable patent literature. However, only a relatively few major types have been reported with full information on their performance. Only those major types of contacting equipment for liquid-liquid extraction fall within the scope of this investigation and are covered in this review.

The results of extensive studies on spray towers (1, 2, 5, 6, 21, 31, 32), packed columns (1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13), wetted-wall towers (7, 12, 13, 14), agitated columns (10, 29), bubble-cap columns (17, 21), horizontal tubes (18), perforated-plate columns (21, 22, 23, 24, 38), and centrifugal extractors (50) have been published.

Limiting flows in spray and packed towers (19, 20, 30), the temperature effect on rate of mass transfer (33), the effect of surface tension on HTU (34, 44), factors influencing the efficiency and capacity of sieve-plate column (38), and liquid-liquid extraction from single drops (2, 32, 35, 37) have also been investigated.

Since Sherwood, Evans, and Longcor (2) found in their study of extraction from single drops that about 40-45 per cent of the solute extracted is accomplished before a drop leaves the nozzle, frequent reformation of droplets of the dispersed phase should greatly improve the efficiency of extraction. A perforated-plate column should achieve this result and a series of investigations on extraction in perforated-plate column were made by various investigators (21, 22, 23, 24, 38).

Later, extraction from single drops was further studied. In the study of the mechanism of solute transfer in spray towers, Licht and Conway (32) reported that the amounts of acetic acid extracted during drop formation using isopropyl ether, methyl isobutyl ketone, and ethyl acetate as solvent are 5, 8, and 17% respectively. These values are, however, less than the values reported by Sherwood, Evans and Longcor. Data on the extraction of acetic acid from benzene drops of known volume by water has been reported by West, Robinson, Morgenthaler Jr., Beck, and McGregor (35), and their results have shown that approximately 14 to 20% extraction was obtained during drop formation.

The rate of emission of droplets through the perforations of the plate is limited by the physical properties of both phases and the size of perforations. Furthermore, each phase only passes once through each plate on its way through the column. It is thus believed that if pulsation were applied to the fluids in the column, so that the fluids would pass back and forth through the same plate before it leaves for the next one, not only the frequency but also the speed of reformation of droplets through each plate will be greatly increased, depending on the frequency and amplitude of the pulsation applied. Dijck (36) was granted a patent on an agitated perforated-plate column for liquid-liquid extraction based on this principle. In Dijck's column, the pulsation was achieved by a reciprocating mechanism which lowered and raised the perforated plates respective to the column and its liquid contents.

Alternately, the plates or the packing may be made stationary and the liquids reciprocated by means of an external piston and cylinder. A preliminary investigation on performance of a pulsed packed column for liquid-liquid extraction was reported by Feick and Anderson (45). Some work on pulsed packed column for liquid-liquid extraction has been conducted by Goundry and Romero (46) under the direction of Wiegandt at Cornell University. Their results have not been published.

Review of Previous Investigations on Liquid-Liquid Extraction in a Perforated-Plate Column.—Investigations have been made by Row, Koffolt, and Withrow (21) on a nine-inch perforated-plate column.

No practical difference was observed between 1/16-, 3/32-, and 1/8-in. diameter holes. These arrangements were found to be more efficient than the spray column and a column packed with copper knitted cloth, but much less efficient than a tower packed with Berl Saddles or Raschig Rings. These investigators also concluded that high rates of flow of the continuous phase have a profound influence on the overall HTU_{ow} results and should be treated separately from lower rates of flow. This effect, however, was minimized by increasing rates of flow of the discontinuous phase and by the use of packing.

The effect of plate spacing on the performance of perforated-plate column was studied by Treybal and Dumoulin (22). Three plate spacings of 3, 6, and 9 inches were used. Decreasing the plate spacing was found to improve the extraction efficiency but to decrease the throughput permissible without flooding. $K_w a$ values for 3-inch spacing are affected by both solvent rates, but for 6- and 9-inch spacings are independent of water rate over the range of flows studied.

A study has been made by Allerton, Strom and Treybal (23) on the extraction rates, holdup, and possible throughputs in the extraction of benzoic acid from toluene and kerosene in a 3 5/8-in. perforated-plate tower. Through-puts were shown to be greatly improved by increasing perforation and down pipe sections. The extraction rates for kerosene with the perforated plates were found to be superior to those obtained in the packed tower, while in the case of toluene they may or may not be better, depending upon the design of the plates.

A 3.75-in. perforated-plate countercurrent liquid-liquid extraction column has been operated by Moulton and Walkey (24) for the extraction of methyl ethyl ketone from mixtures of gasoline using water as the solvent. Overall plate efficiencies for two different plate spacings are reported. The low plate efficiency obtained is attributed to the low degree of agitation existing in the countercurrent liquid-liquid extraction column. At low ratios of solvent rate to feed rate the decrease in efficiency with increase in the solvent-feed ratio is attributed to the decreased contact area caused by a thickening of the hydrocarbon layer beneath each plate. At higher ratios of solvent to feed the increase in efficiency is attributed to the turbulence effect caused by the high solvent rate. This causes the globules of the dispersed phase to decrease in size, thus giving greater contact area with a resulting increase in efficiency.

Review of Previous Investigations on Liquid-Liquid Extraction in a Pulsed Column.—No data was reported by Dijck (36) for his pulsed perforated-plate column.

In the work of Feick and Anderson (45), a 1 7/16-in. column was packed with $\frac{1}{2}$-inch, stainless steel McMahon saddles and 3/8-inch ceramic Raschig rings. The system benzoic acid-toluene-water was used. The column was pulsed by means of a reinforced neoprene diaphragm attached to the bottom section of the column. This was actuated by a rod going to an eccentric which could be adjusted to provide an amplitude of 1/16, 1/8, or $\frac{1}{4}$ inch. Their experiments indicate that a substantial increase in extraction efficiency under

pulsation is indeed observed. The chief effect of the pulsation on extraction efficiency is on the area of contact between the phases. No correlation of data was reported.

Purpose of the Present Investigation.—From the results reported by the previous investigators on perforated-plate towers and pulsed columns, it seems to be very desirable to make an investigation on a pulsed perforated-plate column for liquid-liquid extraction. An improvement on the performance of such column should be expected.

The purpose of this investigation is, therefore, to design a pulsed perforated-plate column in such a way that the actual displacement of fluids in the column caused by pulsation can be accurately calculated, so that the effect of pulsation on the performance of the column can be determined and a general correlation of results can be presented.

The variables investigated were flow rates of both phases, frequency and amplitude of pulsation, and the reversal of phase of dispersion. The effects of these variables on the performance of a pulsed perforated-plate column were studied.

EQUIPMENT AND MATERIALS

A schematic diagram of the extraction unit consisting of perforated-plate column, separators, rotameter, flow controls, pipe connections, pumps, storage tanks, and the other auxiliary equipment is shown in Figure 1. Figure 2 shows a photographic view from the front.

The Extraction Column.—The column itself consisted of 11 segments of pyrex pipe spacers 2-in. inside diameter, 2 5/8-in. outside diameter, and 2-in. long which were manufactured by the Corning Glass Works.

The disengaging chambers at the top and bottom of the column were standard 2-in. cast iron crosses, between which the perforated plates and glass spacers were all held together by four 5/8-inch tie rods. Stainless crosses would be preferable but they were not available at the time. The details of pipe connections to the lower disengaging chamber are shown in Figure 3.

The Perforated Plates.—The brass plates, 5/32-inch thick and perforated with 85 holes 1/16-inch diameter, is shown in Figure 4. The fraction of cross sectional area of the column covered by these 85 holes is 8.51 per cent. The center to center distance between the holes was 3/16 inch. In order to reduce the pressure drop through the plates, all the holes on each plate were countersunk on both sides down to a depth of 1/16 inch (not shown in Figure 4).

The details of sampling device construction are shown in Figure 4. Between the plate and the glass spacer, a layer of 1/8-inch asbestos gasket was used at each joint. A thin layer of graphite was pasted over the surfaces of the gaskets before they were assembled. In all of this work, a plate spacing of 2 inches was used.

The Separators.—The space in the disengaging chambers was not sufficient under most of the operating conditions used in this study to coalesce all the droplets so as to completely separate the two liquid phases. Larger chambers would be more efficient in this respect but they have two rather serious disadvantages. An increase in chamber size would provide more space for mass transfer and thereby obscure the transfer in the column, and also a longer time for the attainment of equilibrium would be required.

In order to avoid these two disadvantages without interfering with the column operation, two separators were used one at each end of the column and in this manner complete separation was obtained. The top separator consisted of a rectangular brass box with a return line for the heavier phase back to the column. Its details of construction and dimensions are shown in Figure 5. In the return line, a mercury seal was used to rectify the flow to one direction. A bottom separator was made of a piece of 1-inch pipe, 4-inch long. It is shown in Figure 3. The separation into two liquid phases at each end of the column was found

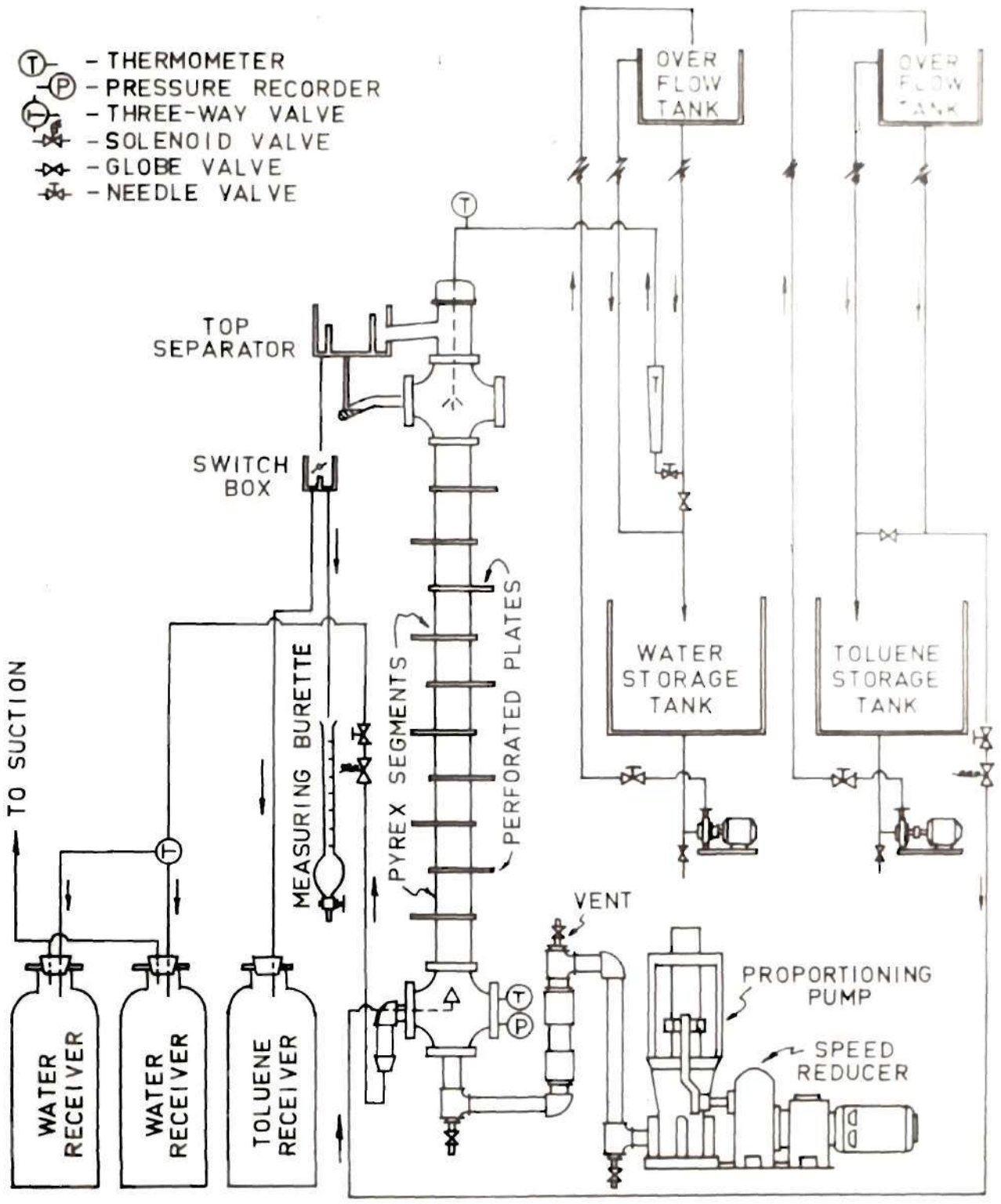


Figure 1. Flow Diagram of Equipment

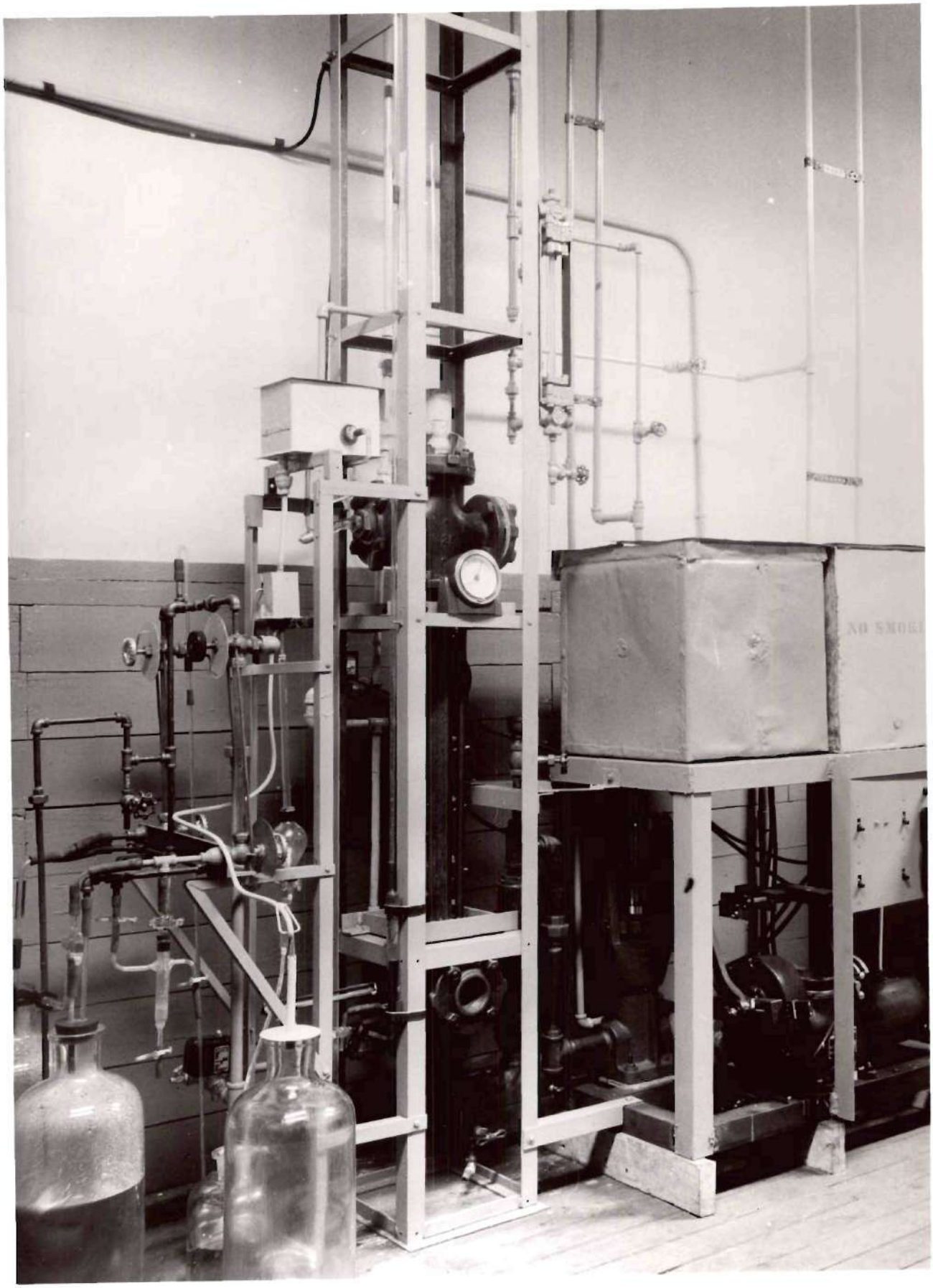


Fig. 2 Liquid–Liquid Extraction Unit

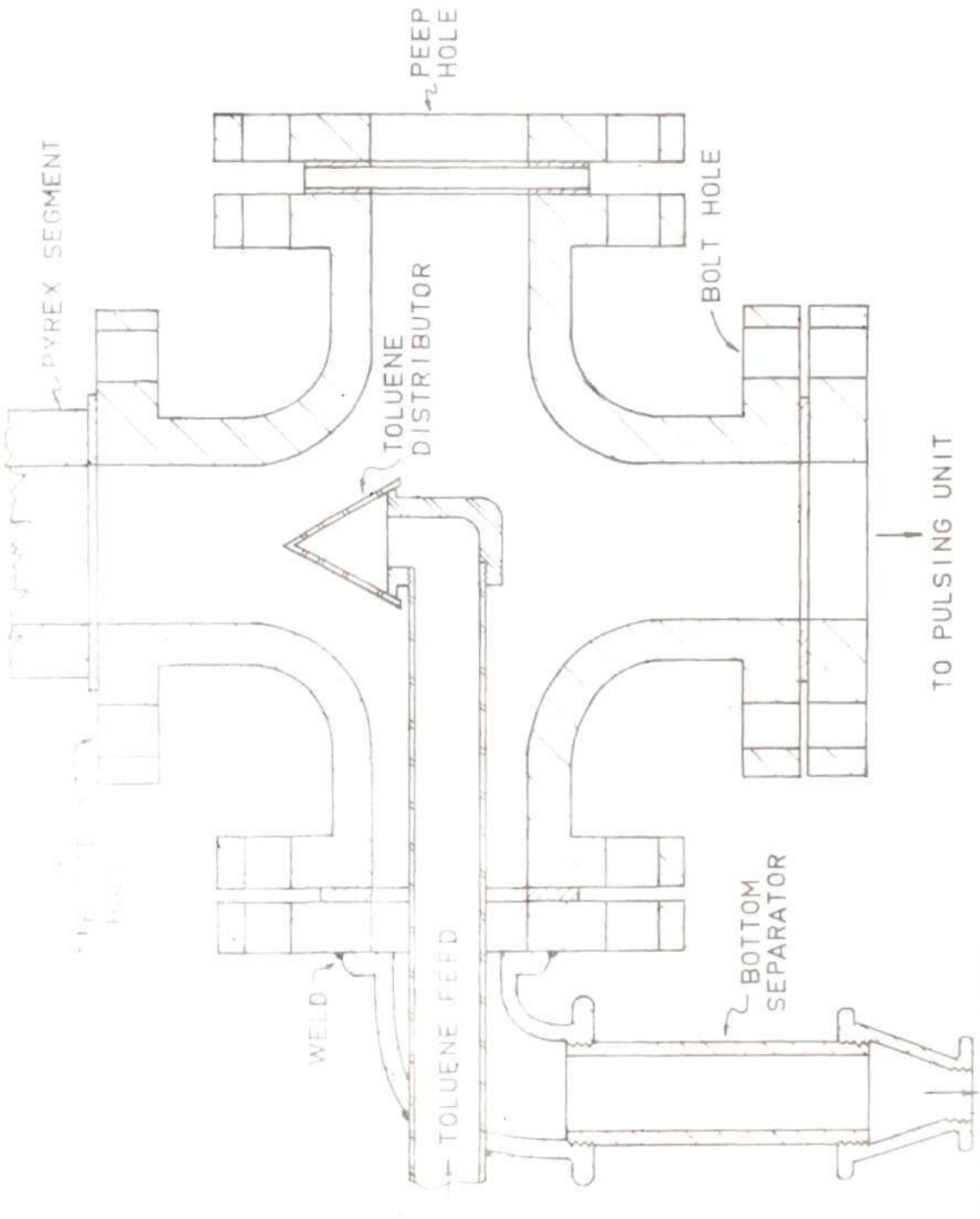


Fig. 3 Bottom Disengaging Chamber and End Fittings

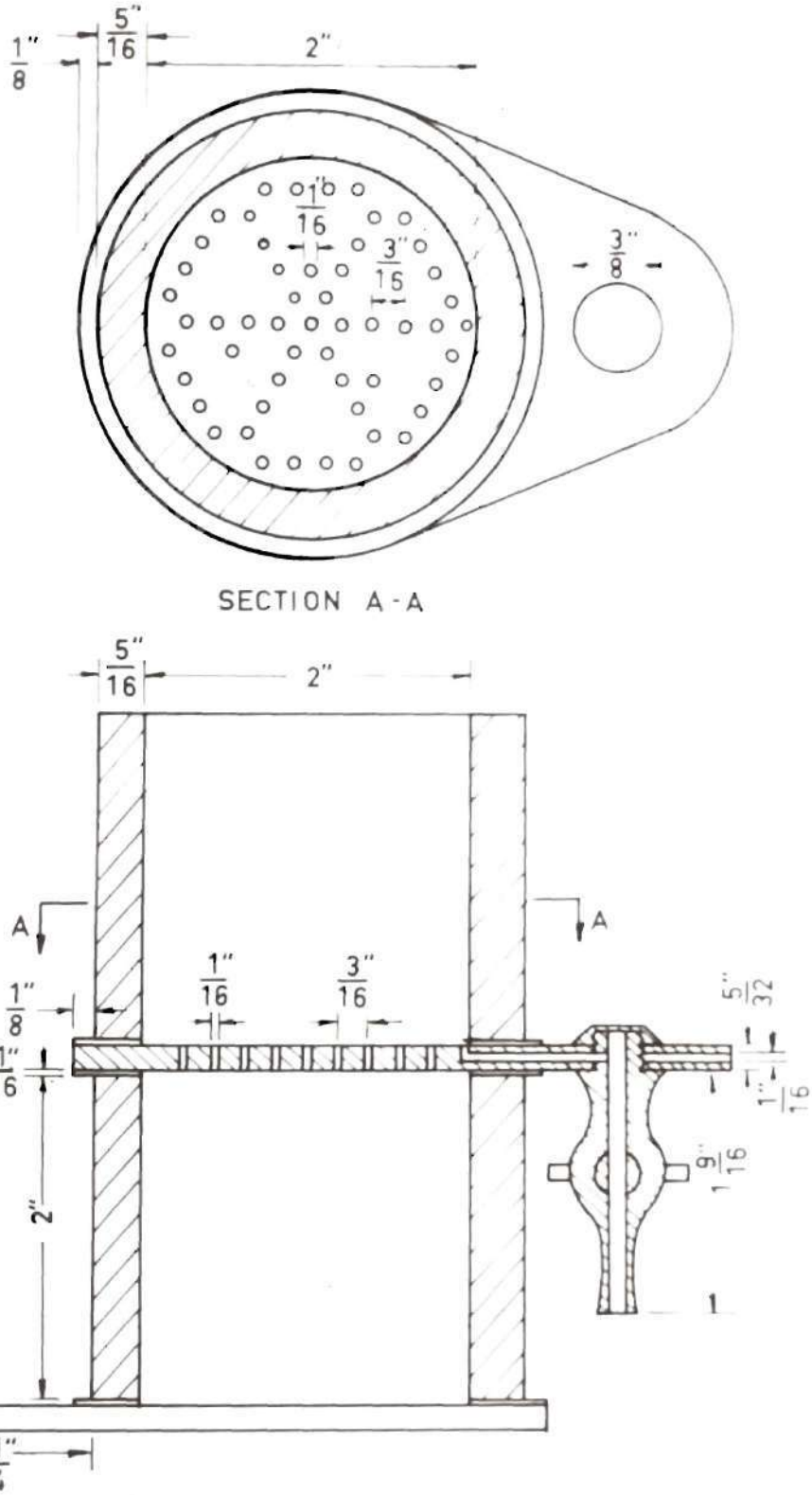
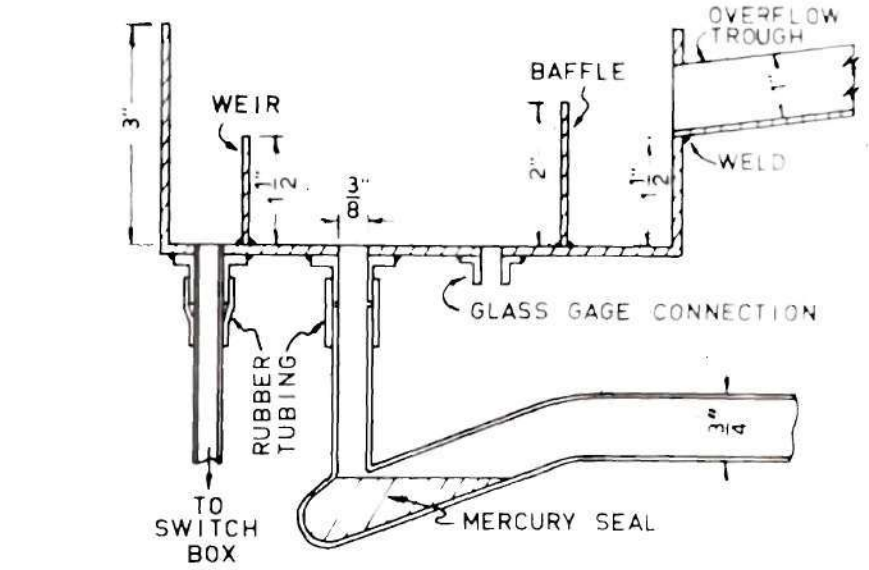
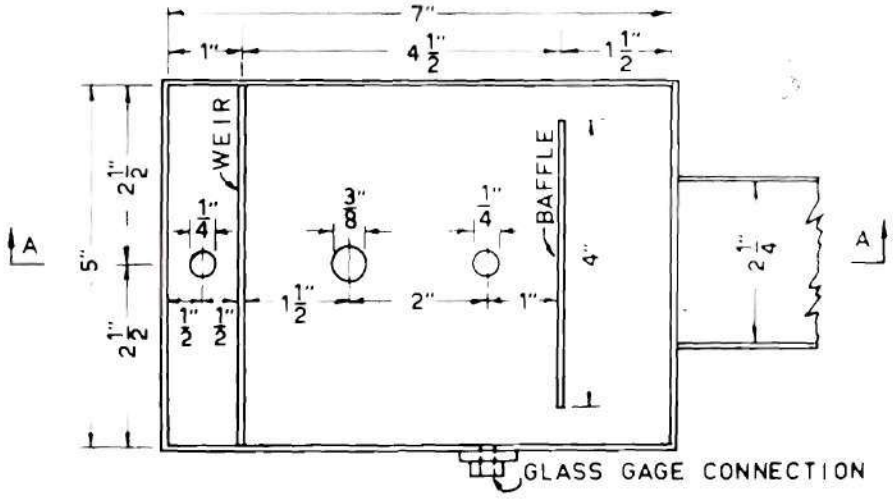


Fig. 4 Details of Plate Constructions and Sampling Device



SECTION A-A



TOP VIEW

Figure 5. Top Separator

to be satisfactory.

The Feed Distributors.—The distributor for toluene feed at the bottom of the column was made of 16-gage aluminum sheet in the shape of a cone whose vertical angle was 60 degrees and a base of $1\frac{1}{2}$ inches in diameter. On the surface of the cone, 78 holes 1/16-inch in diameter were spaced in such a way that their projections on the base would give same number of holes per unit area. In the center of the bottom disengaging chamber, the cone was mounted so that its presence did not restrict the flow of the liquids through the column.

A piece of 3/8-inch stainless pipe projecting down to the center of the top disengaging chamber was used to introduce the water feed.

The Pulse Unit.—The pulsations were generated with a proportioning pump manufactured by Hill-McCanna Company with the following specifications:

Plunger diameter	$2\frac{1}{2}$ inches
Stroke adjustment	by screw adjusting arm
Max. capacity	2.22 GPM at 35 SPM
Min. capacity	0.22 GPM at 35 SPM
Max.pressure	45 psi at 35 SPM
Max. stroke length	$3\frac{1}{2}$ inches

The pump was connected with the motor through a Graham variable speed reducer covering an output speed range from 0 to 110 RPM.

In the U-bend of the 1-inch line connecting the pump and the column, a mercury seal was used to separate the fluid in the proportioning pump, water in this case, from the fluids in the column. A surge section was also provided in the pulsing

line to prevent the possibility of mercury coming over into the pump. The amount of mercury used was carefully adjusted so that it was enough to stop the possible circulation of fluid from pump to the column or vice versa, but not sufficient to fill up the lower part of the bottom disengaging chamber. A vent was provided to drain off any gas entrained in the pulsing line.

Feed Controls.—Two $\frac{1}{2}$-inch solenoid valves, full port type and normally closed, were used to control the streams fed into or taken out of the column. Two sets of specially-made switches for these solenoid valves were mounted on the revolving arm of the proportioning pump so that they were open only when the plunger of the pump came to its top and bottom positions respectively. The opening of the valves was thus synchronized with the strokes of the pump. The duration of opening each valve, as fixed by the length of contact of the switch, was about 7 per cent of the time for a complete revolution. In other words, the volume of displacement by the pump was not appreciably affected by the amount of streams added to or withdrawn from the column; consequently, the volume of displacement in the column due to pulsation could be accurately calculated from the amplitude and frequency of the strokes. A photographic view of the switches for the solenoid valves is shown in Figure 6.

Pressure Recorder.—A special pressure recorder was made of a piece of rubber tubing on which a slot was cut by a sharp blade to a length of approximately 3/8 inch long. One end was plugged

up, and the other was connected with glass tubing to the center of the bottom disengaging chamber. The rubber tubing itself was enclosed in a glass tube, shown in Figure 7. The recorded pressure, therefore, was the maximum pressure at the center of the bottom disengaging chamber including both the pressure to overcome the friction through the plates and the static head of the fluid mixture in the column.

Feed Supplies and Other Accessories.—As shown in Figure 1, a constant head for both water and toluene feed was maintained by circulating the feeds from the storage to two constant level tanks which were placed on the upper floor 10 feet above.

All the lines for toluene solution containing benzoic acid were stainless steel, except the valves. Due to corrosion, copper pipe and brass tanks were not satisfactory for handling toluene solutions of benzoic acid. Glass tanks were used for toluene benzoic acid solution.

The flow rate of water fed into the column was recorded by a rotameter, while that of the toluene solution overflowing from the top of the column was periodically measured in a graduated burette by collecting the stream through a switch box within a fixed time interval.

At the higher water flow rates, it was found necessary to apply suction to the water receiving bottles in order to maintain the desired flow rate of water through the solenoid valve which was closed 93 per cent of the time.

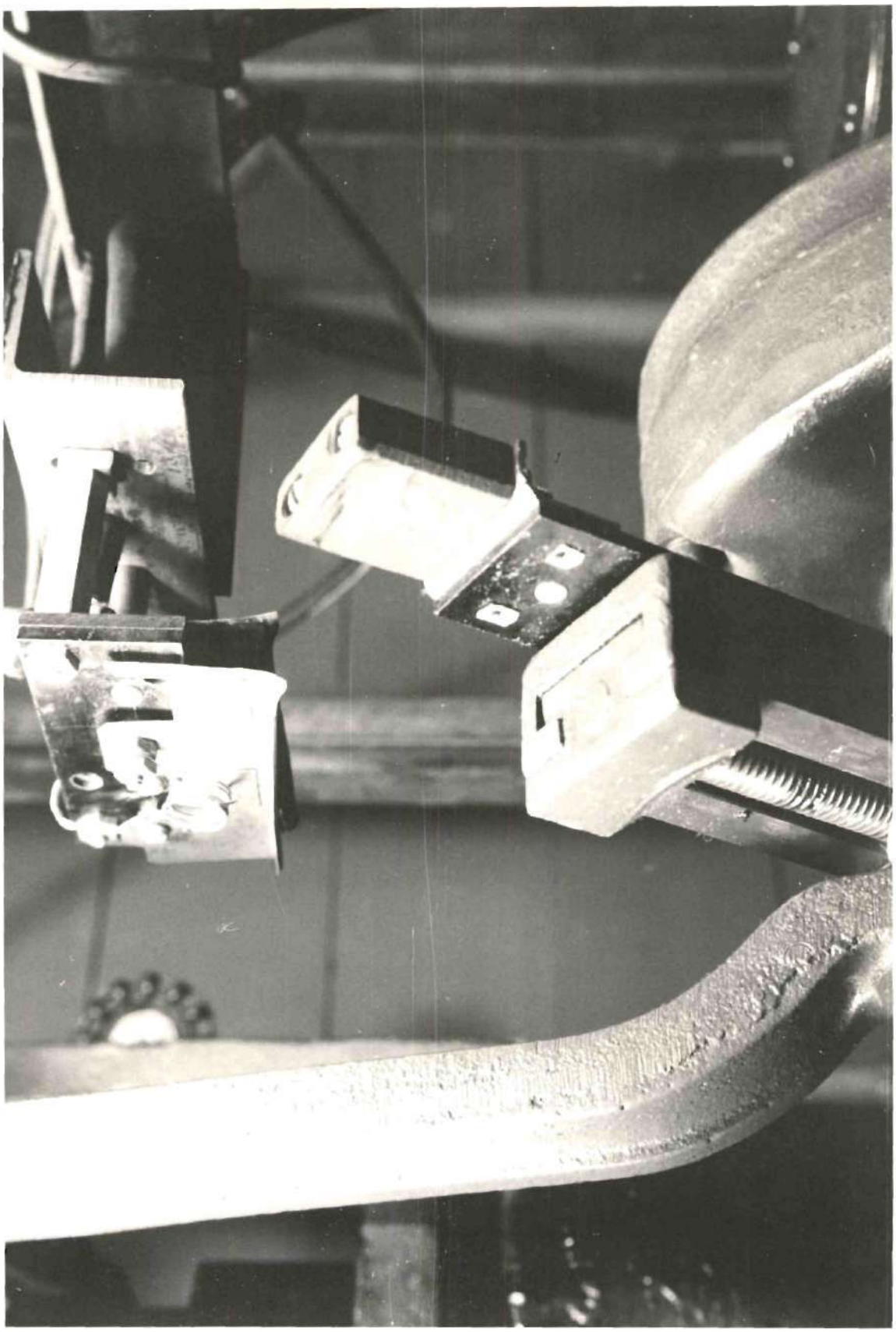


Fig. 6 Synchronizing Switch for Solenoid Valve

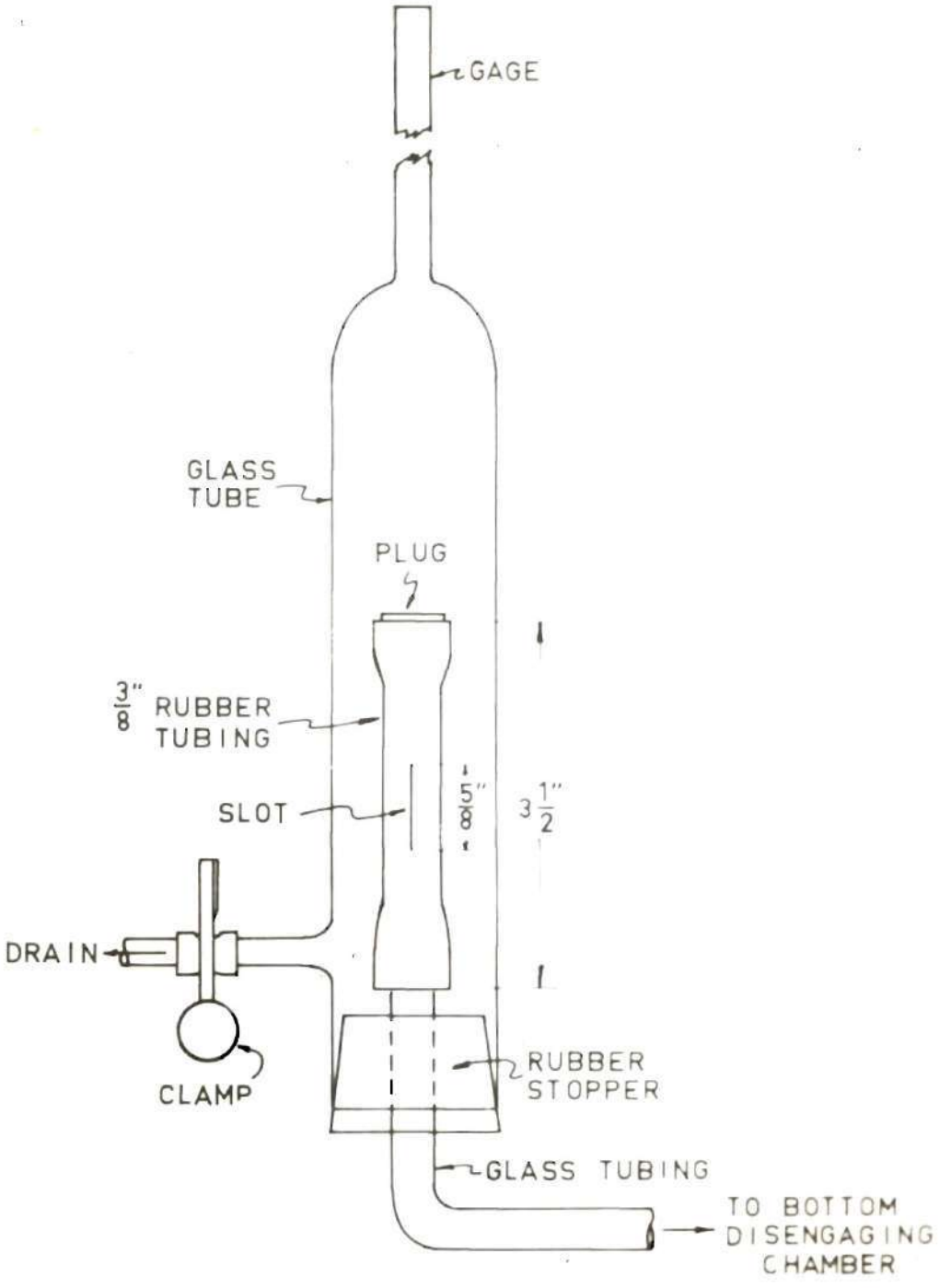


Fig.7 Pressure Recorder

Materials.—Merck's reagent grade toluene, Baker's C.P. grade benzoic acid, and Atlanta city water were used in determining the equilibrium distribution curve and making all the runs. The toluene solution after adding more benzoic acid to make up the proper concentration was used over and over again. The water extract was discarded.

EXPERIMENTAL

Equilibrium Distribution Curve. —Equilibrium distribution on the system benzoic acid-toluene-water was determined for a temperature range from 70 to 91 degrees F. The results were plotted in Figure 8. In the analysis, an alcoholic solution of sodium hydroxide, 0.01N, was used for titration with phenolphthalein as the indicator.

Procedure.—The toluene solution from the previous run was brought up to an approximate concentration of 0.0113 lb. mole per cubic foot by adding more benzoic acid, and fresh water was saturated with toluene by recirculating through a centrifugal pump while a small stream of toluene was introduced into the suction side so that toluene and water were well mixed after passing through the pump. The recirculation was continued after cutting off the toluene stream, until a homogeneous saturated solution was obtained.

Before each run the column and the tanks were well washed and the vent in the pulsing line was flushed with water.

In starting up a run, the column was first filled with a water solution of benzoic acid from the previous run up to the top plate and the rest filled with fresh water. The exact concentration of the initial water solution was immaterial because the purpose of charging the column with this water solution was to hasten the attainment of steady state. Both the fresh water and the toluene solution were then pumped into the overhead con-

stant level tanks, and the regulating valves were set for desired flow rates. The proportioning pump and the switches for the solenoid valves were turned on. By regulating the suction to about 50 inches of water for high water rate, and the valve setting on the exit water line, the level of the interface was set and maintained constant in the top separator for runs with water as the continuous phase. Constancy of the exit compositions of both streams was tested by frequent titration of samples withdrawn at 5-minute intervals. A steady state was reached after a total throughput of three to four times the column volume depending on the flow rates used. For the case with water as the discontinuous phase, the interface was set in the bottom disengaging chamber.

When steady state was obtained, the by-pass for each stream was closed, the electric time recorder was started and the exit streams were collected in the receivers provided for that purpose. At the conclusion of the run the time on the recorder was noted, and the by-pass valves were opened.

During the run, the exit toluene solution was periodically led off through the switch box into a graduated burette where its flow was measured within a fixed time interval. The water rate was indicated by the rotameter which had been previously calibrated (see Figure 9). Since the duration of the run was known, both the water and toluene rates were checked with the total volume of the extract and raffinate collected, thereby providing a check on the precision of the flow measurements.

At the end of a run, the proportioning pump, solenoid valves, and water feed were turned off simultaneously. The liquid mixture in the column then gradually separated into two layers. The toluene layer under each plate did not go through the perforations to the section next above it, because of surface tension and small perforations. After separation, the thickness of toluene layer under each plate was measured and the water samples were withdrawn from each plate for titration. After decanting all the toluene in the top separator, the proportioning pump was then run again with the water feed on, until all the toluene solution in the column rose up and overflowed into a graduated cylinder where its total volume in the column was measured as hold-up. This total volume checked very well with the sum of all layers previously measured under each plate before they were combined.

Concentrations of both exit streams were analyzed by titrating the samples taken from the receivers with 0.01 N alcoholic solution of sodium hydroxide with phenolphthalein as indicator. Blanks on fresh water, which had been saturated with toluene, were also determined. Plate concentrations of toluene layers were not analyzed but calculated by material balance after the concentrations of the water layers were known.

The toluene solution containing benzoic acid was replaced by a fresh batch after every five or six runs. The viscosity and the surface tension of the used batch were determined. Throughout this investigation the viscosity of the toluene feed varied between 0.6230 and 0.6575 centipoise and the surface ten-

sion between 29.5 and 30 dynes/cm., both properties were measured at 80.5 degrees F.

The output RPM of the variable speed reducer and its dial setting were calibrated and the results were plotted in Figure 10. The actual frequency of pulsation was counted by a stop watch, but Figure 10 gives a convenient conversion from desired RPM to dial settings on the speed reducer. The length of stroke was recorded by a tracing device attached to the upper end of the plunger. The volume of displacement of the plunger was checked by observing through the glass column the height of displacement in the column which was partially filled with water. Close agreement of results showed that there was no leakage through the solenoid valves and no air or vapor in the pulsing line.

THEORY

General Theory.—The general principles of mass transfer operations have been successfully applied to liquid-liquid extraction. Using the conventional nomenclature (see page 57), the general rate equations for the system studied are as follows:

$$V'_W dC_W = K(\Delta C)\, dA = K_W a(\Delta C) dV'$$

$$\frac{dC_W}{\Delta C} = \frac{K_W a\, dV'}{V'_W} \qquad (1)$$

Since the system, benzoic acid-toluene-water does not depart appreciably from the simple distribution law in the range studied and the change of concentrations and volume is small, equation (1) can be simplified to

$$K_W a = \frac{N/\theta}{(\Delta C)_m \bar{V}'} \qquad (2)$$

Colburn's HTU method of correlation (25) has been applied by the previous authors (21, 22, 23) to their data on liquid-liquid extraction in perforated-plate columns. The equations may be summarized as

$$n' = \frac{K_W a V'}{V_W} = \frac{K_W a S H'}{V'_W} \qquad (3)$$

$$(HTU)_{OW} = \frac{H'}{n'} = \frac{V_W}{K_W a} \qquad (4)$$

$$(HTU)_T = \frac{V_T}{k_T a} \qquad (5)$$

$$(HTU)_w = \frac{V_w}{k_w a} \qquad (6)$$

From the two film concept, it has been shown that

$$\frac{1}{K_w a} = \frac{H}{k_T a} + \frac{1}{k_w a} \qquad (7)$$

When equation (7) is multiplied by V_w and combined with equations (4), (5) and (6), an useful equation has been obtained by Colburn (25)

$$(HTU)_{ow} = H(\frac{V_w}{V_T})(HTU)_T + (HTU)_w \qquad (8)$$

The application of equation (8) to liquid-liquid extraction data is limited to a certain flow range, because the assumption that $(HTU)_T$ and $(HTU)_w$ are independent of flow rates of both phases is not correct, especially in the region where high water rate is involved.

Equation (8) was modified by Colburn and Welsh (8) in their study of individual film resistances in liquid-liquid extraction into

$$(HTU)_{oc} = C_2(\frac{W_c}{W_d})^{.75} + C_1(\frac{W_c}{W_{dm}}) \qquad (9)$$

Equation (9), when converted with the nomenclature listed on page58, becomes

$$(HTU)_{ow} = C_2(\frac{V_w}{V_T})^{.75} + C_1(\frac{V_w}{V_T})H \qquad (10)$$

Film HTU's were also correlated by Colburn and Welsh (8) with the equation

$$(HTU)_c = C_2\left(\frac{W_c}{W_d}\right)^{.75} \tag{11}$$

which can be converted with similar nomenclature to

$$(HTU)_w = C_2\left(\frac{V_w}{V_T}\right)^{.75} \tag{12}$$

If equations (10) and (12) are combined, the following equation is obtained

$$(HTU)_{ow} = C_1\left(\frac{V_w}{V_T}\right)H + (HTU)_w \tag{13}$$

By comparing equations (8) and (13), it is noted that they are the same if $(HTU)_T$ in equation (8) is assumed to be constant.

Similar correlation with equation (11) was reported by Laddha and Smith (28) in their investigation on film resistances in liquid-liquid extraction with a two-component system.

Derivation of Equations for Correlation of Present Data.—Dimensional analysis has been widely used in correlating results on heat, mass, and momentum transfers. This method is particularly valuable where the mathematical relations are unknown or complex and will indicate the logical grouping of the factors into dimensionless combinations. The latter feature is helpful in interpreting data where two or more factors have been varied in different experiments.

The superimposition of a pulsation on a perforated-plate column for liquid-liquid extraction would make an analytical analysis exceedingly difficult, and since at the present time

not enough is known about the factors affecting a pulse column to make such an analysis, a dimensional study will be made in this investigation. The Reynolds number through the perforations will be used to characterize the pulsation in the column.

Mass transfer film coefficients in wetted-wall columns have been shown by Gilliland and Sherwood (47), and Chilton and Colburn (48) to be functions of Reynolds and Schmidt numbers. Dodge and Dwyer (27), and Brinsmade and Bliss (16) reported that mass transfer film coefficients are affected by the flow rates of both phases. The mass transfer film coefficients for liquid-liquid extraction in a pulsed column under constant temperature are then functions of toluene rate V_T, water rate V_W, diameter of the perforation D, mass velocity G through the perforation, density ρ, and viscosity μ of the liquid mixture. The density and the viscosity of the liquid mixture passing through the perforations depend not only on temperature but also on the volume ratio of toluene to water in the mixture, therefore the terms ρ and μ are retained in the analysis.

$$k = \phi' (V_T, V_W, D, G, \rho, \mu) \qquad (14)$$

Expressing the variables in terms of mass, length, and time

$$L\theta^{-1} = \phi'(L\theta^{-1})^n(L\theta^{-1})^m(L)^d(ML^{-2}\theta^{-1})^l(ML^{-3})^e(ML^{-1}\theta^{-1})^f$$

$$\Sigma M \quad 0 = l + e + f$$

$$\Sigma\theta \quad -1 = -n-m-l-f$$

$$\Sigma L \quad 1 = n+m+d-2l-3e-f$$

Retaining the exponents n, m, and l

$$k = \phi'(V_T)^n(V_W)^m(D)^{-1+n+m+l}(G)^l(\rho)^{n+m-1}(\mu)^{1-n-m-l}$$

Rearranging

$$kD = \phi'\left(\frac{DV_T\rho}{\mu}\right)^n\left(\frac{DV_W\rho}{\mu}\right)^m\left(\frac{DG}{\mu}\right)^l \qquad (15)$$

From the results of the present investigation it was found that holdup, which is defined as the volume percentage of the dispersed phase in the toluene-water mixture, varied from 5 to 48 per cent. A corresponding change of ρ/μ ratio was found by calculation to be approximately 4 per cent. Since the ratio ρ/μ varied about 4 per cent in the whole range of variation of the composition of the holdup, and since ρ and μ appear in the analysis as the ratio ρ/μ, an average value of holdup of 16% toluene was used to calculate the values of ρ and μ for the liquid mixture flowing through the perforations. In as much as the diameter of the perforation is fixed, equation (15) can be simplified into

$$k = \phi''(V_T)^n(V_W)^m\left(\frac{DV\rho}{\mu}\right)^l$$

or

$$k = \phi''(V_T)^n(V_W)^m(Re)^l \qquad (16)$$

During the course of the present investigation, it was observed that the drop size of the dispersed toluene was not uniform. Measurement of drop diameter was not attempted. The interfacial area between two liquid phases could not be calcu-

lated although the volume percentage of toluene in the liquid mixture was known, and therefore it is combined together with the transfer coefficient k. This procedure has been commonly used. Equation (16) can be written as

$$ka = \phi''(V_T)^n(V_W)^m(Re)^l \tag{17}$$

When equation (17) is applied to both toluene and water film coefficients, it becomes

$$k_Ta = \phi''_T(V_T)^n(V_W)^m(Re)^l \tag{18}$$

and

$$k_Wa = \phi''_W(V_T)^t(V_W)^s(Re)^r \tag{19}$$

The exponents l and r have been found to be functions of (52) the geometric shape of the equipment. Consequently, in this study these exponents will be assumed to be equal.

Equations (7), (18) and (19) may be combined to give

$$\frac{(Re)^l}{K_WaH} = \frac{1}{\phi''_T(V_T)^n(V_W)^m} + \frac{1}{H\,\phi''_W(V_W)^s(V_T)^t} \tag{20}$$

Brinsmade and Bliss (16) pointed out that either individual coefficient can be affected by both fluid rates, but not both coefficients simultaneously. Furthermore, the water film resistance for the system benzoic acid-toluene-water is much smaller than that of the toluene film as reported by Allerton, Strom and Treybal (23). The exponent t in equation (20) can be assumed to be zero. With constant flow rate of water phase,

equation (20) can then be written as

$$\frac{(Re)^{l}}{K_w a H} = \frac{1}{\phi_T (V_T)^n} + \frac{1}{H \phi''_w (V_w)^s} \qquad (21)$$

If $\frac{(Re)^{l}}{K_w a H}$ is plotted vs. $\frac{1}{(V_T)^n}$ with the water rate held constant, the intercept is $\frac{1}{H \phi''_w (V_w)^s}$ and the slope is $\frac{1}{\phi_T}$. This is the familiar Wilson plot, only in a modified form. Henry's constant H in the previous derivation can be omitted if temperature changes involved are not too large. However, in this work the maximum variation of H was 7 per cent, and therefore this term is retained.

Rearranging equation (21),

$$\frac{(Re)^{l} V_w}{K_w a} = \frac{H V_w}{\phi_T (V_T)^n} + \frac{1}{\phi''_w (V_w)^{s-1}} \qquad (22)$$

or $$(HTU)_{ow}(Re)^{l} = \frac{H V_w}{\phi_T (V_T)^n} + \phi'_w \qquad (23)$$

where $$\phi'_w = \frac{1}{\phi''_w (V_w)^{s-1}}$$

It is evident from equations (5) and (6) that unless $k_T a$ and $k_w a$ are directly proportional to their respective toluene and water rates, $(HTU)_T$ and $(HTU)_w$ should vary with the flow rates. Since the individual film coefficients are not directly proportional to the flow rates, a plot of $(HTU)_{ow}$ versus $H(\frac{V_w}{V_T})$ (equation 8) is not strictly correct, although it may be applied without appreciable error within certain flow ranges. According to equation (23), however, a plot of $(HTU)_{ow}(Re)^{l}$ against $\frac{H V_w}{(V_T)^n}$

under constant water rate is theoretically correct, giving the intercept $\frac{1}{\phi''_w (V_w)^{s-1}}$ and the slope $\frac{1}{\phi_T}$.

Empirical Correlation.—Sherwood and Holloway (26) reported the following equation representing the data of Borden and Squires (49) on gas absorption

$$K_G a = \alpha G^n L^m$$

This empirical equation was also used by Dodge and Dwyer(27) to correlate their data on gas absorption. A similar empirical equation can be written for liquid-liquid extraction in a pulse column

$$\frac{K_w a H}{(Re)^t} = \phi''_T (V_T)^n (V_w)^m \qquad (24)$$

Equation (24) can also be derived from equation (20) by neglecting the water film resistance.

CALCULATIONS

In this study all runs for which the error in the material balance was greater than 5% were discarded.

Values of $K_W a$, $k_T a$, $k_W a$, $(HTU)_{OW}$, $(HTU)_T$ and $(HTU)_W$ were computed from equations (2), (18), (7), (4), (5), and (6) respectively. The effective volume used in the calculation of $K_W a$ values from equation (2) was 0.1015 cubic foot, which included the disengaging chambers, the top separator and the glass column.

The slope dC_T/dC_W of equilibrium curve at different temperatures was taken at an average toluene concentration. For each run this slope was determined according to the temperature. Henry's constant H, the reciprocal of slope, is equal to dC_W/dC_T.

The Reynolds number through the perforations was calculated using the density and viscosity of a liquid mixture containing 16 per cent toluene by volume which was the average analysis of the holdup of all the runs. Although a rigorous calculation of ρ and μ using the actual holdup analysis for each run was possible, nevertheless the variation in holdup analysis has but little effect upon the ratio ρ/μ, since both ρ and μ decreased with increasing holdup. For the same reason, an average of room temperatures of 80 degrees F. was used to calculate ρ/μ for all the runs. The term V, the arithmetic average velocity of liquid mixture through the perforations, was calculated from the displacement of the pump and the area of perforations on the plate.

RESULTS AND DISCUSSION

Unique Characteristics of a Pulsed Column.—With constant flow rates of both phases, the effect of pulsation expressed in terms of Reynolds number Re on the rate of mass transfer is shown in Figure 11. Each curve shows a sharp break in slope at a Reynolds number between 1050 and 1200, which divides the curve into two regions. The explanation for this transition from one region to the other will be discussed later. The lower region, where the effect of Reynolds number on $K_w a$ values is small, is called the streamline region and the other the turbulent region.

With varying flow rates in both phases, the slopes of both streamline and turbulent regions remain unchanged which is shown by curves A, B, and C in Figure 11. The critical Reynolds number, however, decreases slightly with increasing flow rates.

When operating near the lower end of the stream region, the water phase in the space between plates was broken up during the down stroke of the plunger into large droplets which were separated from one another and remained separated during the up stroke. The water droplets appeared in a "cell-like" formation which was stabilized by a thin film of toluene as shown in Figure 12. The reason for this cell formation phenomenon is believed to be due to the fact that, during the down stroke, water phase was sucked downward through the perforations against a thin layer of toluene accumulated underneath the plate and expanded into drops surrounded by toluene film which has low surface tension. This phenomenon is similar to the formation of foams formed by blowing air into a

soap solution.

The toluene phase in the cell-like mixture began to coalesce into droplets after leaving the top plate and rose up as a dispersed phase in the top disengaging chamber. In the streamline region, therefore, the water phase was actually discontinuous between the plates but continuous in the top and bottom disengaging chambers. To avoid confusion with the case to be described later where water was the true dispersed phase throughout the column, the water phase, despite its cell-like formation, was still considered as continuous in this streamline region.

As the Reynolds number increased in the streamline region, the cell size became smaller and the cell formation phenomenon gradually disappeared (figure 13). Upon further increase of Reynolds number beyond 1200, the toluene phase became clearly dispersed with vigorous turbulence in the water phase as shown in Figure 14.

The change from streamline to turbulent region was not abrupt. A buffer zone around Reynolds number 1000 was observed, where a mixture of both cell formation and turbulent dispersion could exist.

As the Reynolds number was further increased, the size of the dispersed droplets became finer and finer, until with a Reynolds number around 1800 the entrainment of fine toluene droplets in the exit water stream could not be settled out in the bottom separator. With all toluene rates and for all water rate investigated, flooding occurred at a Reynolds number of

approximately 1800. Curves in Figure 11, therefore, should not be extrapolated at the upper end.

When operating at the lower end of the streamline region with a Reynolds number around 400, the column was only partially filled with cell-like dispersions, leaving the lower space of each column segment mainly full of continuous water phase, Figure 12. The column seemed to be underloaded. For this reason, runs with Reynolds number less than 400 were not attempted.

When pressure drop across the column was plotted versus Reynolds number in Figure 15, the same shape of curve was obtained as in Figure 11, with the same critical Reynolds number (around 1200). Since the water phase was fed into the column at the top, its flow rate did not affect the pressure drop, which however was found to be slightly increased by increasing the toluene rate.

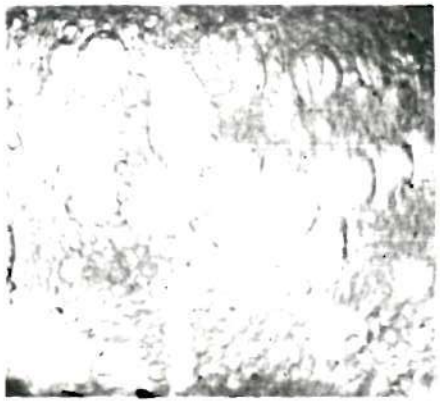
To study the cause of the break of the curve in Figure 11 and 15, the column was operated with water alone under the same conditions as operating with water and toluene. Water fed into the bottom of the column through the toluene feed line was kept at a rate of 40.2 cu. ft. per sq. ft. per hour for all the following runs. A set of pressure drop data was taken with varying Reynolds numbers and these are recorded in Table I.

Table I. Pressure Drop with Water Phase Alone

Stroke Length Inches	Frequency RPM	Re	Pressure Drop Inches of Water
1.44	9.75	425	61.4
1.44	27.50	1197	75.3
1.44	30.4	1325	80.5
1.44	37.1	1615	89.3

Test Run Re = 390

Run No. 41 Re = 417



Run No. 44 Re = 535

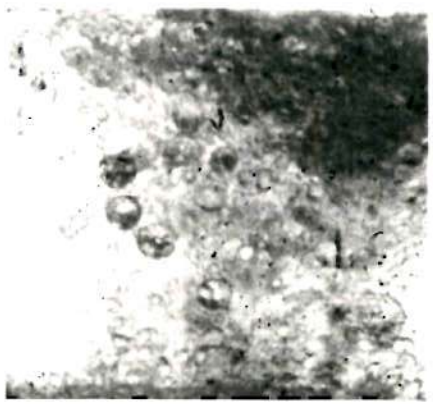
Fig. 12 Dispersion of Toluene in Lower Streamline Region

Run No. 43 Re = 750

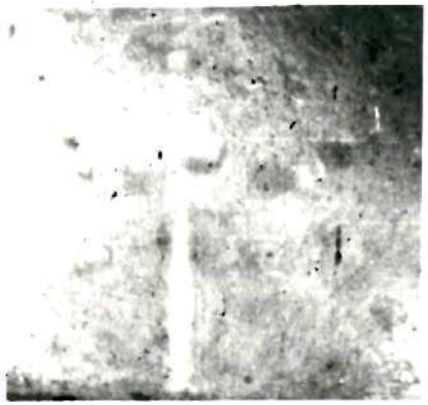
Run No. 42 Re = 944

Run No. 35 Re = 1029

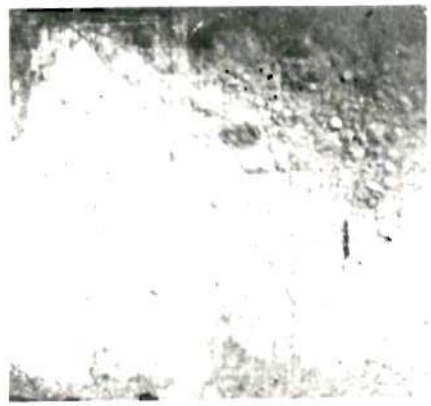
Fig. 13 Dispersion of Toluene in Upper Streamline Region



Run No. 40 Re = 1197



Run No. 37 Re = 1313



Run No. 39 Re = 1475

Fig. 14 Dispersion of Toluene in the Turbulent Region

Run No. 47 Re = 943

Fig. 27 Dispersion of Water in the Streamline Region

These data, when plotted in Figure 15, showed the same shape of curve with a break at a Reynolds number around 1200. It is evident that the break of curves in Figure 11 and 15 is due to the change of nature of the flow of liquid mixture in the column; that is, from streamline flow to turbulent flow.

Consider a drop issuing from a nozzle pointing upward. As pointed out by Hayworth and Treybal (37), there are four forces acting on the drop; namely, kinetic force F_k and bouyant force F_B pushing it away from the nozzle, while its surface tension F_σ and the resistance F_R of a slow moving pool of fluid around the nozzle are retarding it. In streamline flow, F_k and F_R are comparatively small and the stream retains its laminar nature after leaving the plate. In turbulent flow with F_k and F_R greatly increased, a fast moving stream from the nozzle or perforation is ejected into a comparatively slow moving fluid, resulting in a large loss of kinetic energy which is in turn dissipated into the chaotic motion, that is called turbulence. This transition from streamline to turbulent flow explains why the curves break in Figures 11 and 15. The critical Reynolds number at which the transition takes place depends on the geometrical shape of the nozzle or the conduit through which the fluid is passing.

When the velocity through the nozzle is zero, both F_k and F_R become zero. Then whether the drop will detach from the nozzle or not depends on the relative magnitude of the forces F_B and F_σ ,

where $F_\sigma = \pi D \sigma$

and $F_B = V_\sigma \Delta \rho g$

If $\pi D \sigma$ is greater than $V_\sigma \Delta \rho g$, the drop will not detach from the nozzle. This explains why the toluene layer underneath the plate did not penetrate through the plate after separation as mentioned on page 25.

Effect of Varying Pulsation.—It is seen from Figure 11 that the slopes are 0.175 and 1.07 for the streamline and turbulent regions respectively. The mass transfer coefficients are greatly increased by operating in the turbulent region. Similarly, the pressure drop across the column is increased in a same manner as the mass transfer coefficients, with slopes 0.155 and 0.85 for streamline and turbulent regions respectively.

The similarity of the shape of the curves in Figures 11 and 15 leads to two following deductions:

(1) The rate of mass transfer in the pulse column was a function of friction loss in the column. This seems to agree with the Reynolds analogy. More vigorous turbulence of fluid mixture with faster scraping action of films between phases caused higher pressure drop across the plates and gave higher rates of mass transfer.

(2) Most of the mass transferred from one phase to another was accomplished while both fluids were emerging from the perforations and very little occurred between the plates. This was especially true in the streamline region. In other words, the

transfer of mass carried out in the space between plates is small compared to that accomplished by the action of the plates.

For the data shown in Figure 11, different stroke length and frequencies were used. The effect of pulsation on transfer coefficients was found to be independent of stroke length or RPM, and dependent only on the product of the two. For practical consideration, low frequency and stroke length seem to be more preferable. However, a constant interphase was more easily maintained when operating under short strokes. In this work a stroke length of 1.44 inches or a height of 2.25 inches of displacement in the column was found to be satisfactory. In other words, there was a linear displacement of little over one plate spacing.

Effect of Varying Flow Rates.—With a constant Reynolds number of 943, a series of runs was made to study the effect of flow rates of both phases on overall mass transfer coefficients. The results plotted in Figures 16 and 17 show that the overall mass transfer coefficient was only slightly affected by the water rate ($K_w a = C_1(V_w)^{0.11}$), but considerably more by the toluene rate ($K_w a = C_2(V_T)^{0.55}$) in the streamline region. In the turbulent region, the overall transfer coefficient seems to be even less affected by the water rate while the toluene rate has a large influence upon this coefficient as shown in Figure 18.

Figure 19 is the orthodox plot of $K_w a$ versus V_T and shows that even with a three fold variation in the water rate all the data falls on a straight line. It is evident that the effect

of the water rate on overall transfer coefficient was negligible compared to that of the toluene rate.

As pointed out by Colburn (25), the optimum value of HV_W/V_T for practical operation of commercial columns would be in the order of 0.5 to 0.8. The range covered in this work was 0.02 to 0.13. If the ratio of V_W/V_T is increased in order to reach the optimum range, the concentration of benzoic acid in the exit toluene solution becomes so low that the assumption of straight line relationship in the lower region of the equilibrium distribution curve can not be made. Higher values of HV_W/V_T beyond 0.13, therefore, were not attempted.

HTU Correlations.—In the previous investigations (21, 22, 23), the results on liquid-liquid extraction in a perforated-plate column were correlated by Colburn's HTU method (25) (equation 8). A similar plot, Figure 20, was made with the present results. HTU_W, the intercept, and HTU_T, the slope, are apparently decreasing as the Reynolds number increases. In other words, both film resistances become smaller with the increase in the Reynolds number.

Another fact is clear from Figure 20: that Runs No.5, 6, and 30 whose flow rate ratio of water to toluene was high, do not fall on the straight line.

The various curves in Figure 20 have Reynolds numbers as a parameter. This data falls more or less on one curve (Figures 21 and 22) when $(HTU)_{OW}(Re)^l$ is plotted against $H(\frac{V_W}{V_T})$. The values

of l (.175 for the streamline and 1.07 for the turbulent region) may be found in Figure 11. While several points do not lie on a straight line, the correlation is better than Colburn's correlation shown in Figure 20.

As pointed out in the section on theory, the plots in Figures 20, 21, and 22 are limited by the assumption that HTU_W and HTU_T are independent of flow rates. This assumption apparently does not hold in the region of low toluene rate and high water rate. Row, Koffolt, and Withrow (21) reported that "there was marked deviation from straight line with low toluene rate and high water rates of flow but the curvature had a tendency to straighten out as the toluene rate of flow was increased. These deviations suggested that other factors besides flow rates affect the HTU values". This explanation seems to be wrong, since according to equation (21), the deviation from straight line relationship in Figures 21, 22, and 23 should be expected.

The results were then correlated by equation (21). By trial and error, the best straight line was obtained when a value of n equal to 0.67 for the streamline region was used (Figure 23). The intercept $\frac{1}{\phi''_W (V_W)^{s-1}}$ is 0.55 and the slope $\frac{1}{\phi_T}$ is 33.1. It can be seen that all data fall on the straight line in Figure 23. Similarly for the turbulent region, the value of n was found to be 1.25, and the results are shown in Figure 24, giving an intercept 550 and a slope 136,500.

In the determination of equation (21), the assumption of constant water rate was made. Strictly speaking a series of lines

should be drawn in Figures 23 and 24 with each line representing one constant water rate. However, since all points within the range of water rates studied fell so close to the same straight line, it is obvious that such procedure is neither practical or necessary. The intercept and the slope of the curves in Figures 23 and 24 were, therefore, obtained using an averaged water rate.

Resolution of Overall Coefficient into Individual Film Coefficients.- By comparing equations (20) and (21), it is seen that

$$\phi_T = \phi''_T \, (V_W)^m \qquad (25)$$

Since ϕ_T is obtained from an averaged water rate in Figures 23 and 24, equation (18) can be written with approximation as

$$k_T a = \phi_T \, (V_T)^n (Re)^l \qquad (26)$$

Since the values of ϕ_T, n, and l have been found from previous plots, $k_T a$ can be calculated. After determining $k_T a$, the values of $k_W a$, $(HTU)_T$, and $(HTU)_W$ can be obtained from equations (7), (5), and (6) respectively. As an illustration, few runs were taken for such calculations, and their results are summarized in Table 2.

Table 2 shows the relative magnitude of film coefficients. $k_W a$ is about 100 to 200 times larger than $k_T a$ depending on the flow rates, and therefore the toluene film is controlling. This fact was repeatedly noticed in the previous plots.

It is clear from the results of Run No.30 in Table 2 that the value of HTU_W is appreciably affected when the ratio of water

Table 2.

Resolution of Overall Coefficient into Film Coefficients

Run No.	$\frac{1}{\phi_T}$	V_w	V_T	$V_T^{.67}$	$(Re)^{.175}$	$K_w a$
4	33.1	20	39.5	11.75	3.32	22.4
7	33.1	38.4	20.8	7.64	3.32	15.5
8	33.1	38.4	28.8	9.51	2.88	17.5
13	33.1	38.4	29.7	9.70	3.36	20.5
22	33.1	37.2	33.3	10.70	3.31	19.7
30	33.1	58.9	20.4	7.55	3.32	17.4
43	33.1	59.4	48.5	13.5	3.18	27.4

Run No.	H	$k_T a$	$k_w a$	HTU_T	HTU_w
4	o.o434	1.18	128	33.5	0.156
7	0.0421	0.765	107	27.2	0.357
8	0.0429	0.827	192	34.8	0.20
13	0.0429	0.984	208	29.9	0.185
22	0.0413	1.07	82	31.1	0.454
30	0.0429	0.757	1250	27.0	0.047
43	0.0429	1.30	294	38.2	0.202

to toluene rates is high. The abnormally high value of $k_w a$ is obviously due to a large reduction of water film resistance. This fact also explains the deviation of straight line relationship in Figures 20 and 21.

Empirical Correlation of Results.—Equation (24) can be considered as empirical or as derived from equation (20) by neglecting the water film resistance, which was found to be very small. The results were correlated by equation (24), and are shown by Figure 25. All the data fall nicely on a single straight line. Neither of the curves in Figure 25 can be extrapolated because of the previously discussed limitations of flooding and of underloading conditions in the column.

When the water film resistance is negligible, equation (21) can be applied if the second term on the right is dropped. The results of this procedure were plotted in Figure 26. The alignment of data on straight lines is as good as that obtained from empirical equation (24). The difference between the plots in Figures 25 and 26 is due to the retainment of the term $(V_w)^m$ in equation (24) as compared to equation (21) without the second term on the right. The omission of the water film resistance is again justified. In any system where one film resistance can not be neglected, equation (21) must be used.

Water as Dispersed Phase.—Treybal and Dumoulin (22) have attempted, but not successfully, to use water as the discontinuous phase in a perforated-plate column. The reason for this lack of success was

stated to be that "water goes through the perforations, and then wets the underneath side of the plates and either drops down in blobs or runs over to the side of the tower and flows down in a film". In the present work no difficulty was encountered in operating the column with water as discontinuous phase when pulsations were applied.

In the operation with water as discontinuous phase, the interface was maintained in the bottom disengaging chamber, and all the other operating conditions remained same. The droplet size of dispersed water phase was found to be small (Figure 27).

The results with water as dispersed phase were plotted in Figures 19 and 20. Figure 19 shows that, with a high toluene rate above 15 ft.^{3}/hr.ft.^{2}, a lower transfer coefficient was obtained by dispersing water in toluene, but at low toluene rate below 15 ft.^{3}/hr.ft.^{2} a higher transfer coefficient was obtained. Whether the water or toluene phase should be dispersed in order to obtain the highest transfer coefficient will consequently depend on the toluene rate used.

As pointed out previously, if the column is operated on a commercial basis with a value of $H(\frac{V_W}{V_T})$ between 0.5 to 0.8, a large ratio of water to toluene would be used. With the toluene rate of 15 ft.^{3}/hr.ft.^{2}, a minimum water rate of 174 ft.^{3}/hr.ft.^{2} would be necessary. Such a high water rate was not covered in this work, nor has it been reported in the literature for perforated-plate columns, and therefore whether or not this high water rate would flood the column is not known at the present time. If flooding should occur, then the toluene rate must be cut down to a value

below 15 ft.^{3}/hr.ft.^{2}, and water should be used as the discontinuous phase so as to obtain the highest transfer coefficients. This agrees with the general rule pointed out by Colburn and Welsh that the phase with higher flow rate is generally made discontinuous.

To explain the low values of transfer coefficients with water as discontinuous phase and a toluene rate above 15 ft.^{3}/hr.ft.^{2}, it may be said that the increase of water film resistance more than offsets the decrease of toluene film resistance caused by the phase reversal.

From Figure 20, the intercept $(HTU)_W$ and the slope $(HTU)_T$ were taken from the line with water as the dispersed phase and included in Table 3 for comparison with published data on perforated-plate columns.

Data with water dispersed in toluene phase in perforated-plate column are not available in the literature. However, data with water dispersed in toluene phase in a packed column were reported by Appel and Elgin (5), and their $k_W a$ values are slightly lower than that found in this investigation.

Comparison of Results from the Literature.—Only the results on perforated-plate columns using the system benzoic acid-toluene-water were taken from the literature for comparison. But still, due to wide variation of conditions under which the results were obtained by different investigators, only a rough comparison of results to show relative magnitudes is possible.

Since the values of $(HTU)_W$ and $(HTU)_T$ reported in the literature were obtained as intercepts and slopes from plots similar to Figure 20, the same procedure was followed in obtaining $(HTU)_W$ and

Table 3. Comparison of HTU's in Perforated-Plate Columns.

Plate Characteristics	Reference	Continuous Phase	Dispersed Phase	$Ft.^3/hr.ft.^2$ Range of flow rates covered V_W	V_T	HTU_W	HTU_T
6-in spacing-holes	3/32-in. Row, 1/8-in. holes	(21) Koffolt & Withrow	Water	Toluene	11.8-37.1	12.5-47.7	0.25	106
			"	"	11.8-37.1	11.8-47.7	0.25	115
3/16-in. holes	3-in. spacing	(22) Treybal &	"	"	15.1-32.5	9.7-81.8	0.65	30
	6-in. spacing	Dumoulin	"	"	22.9-41.5	30.2-76.8	0.25	63
	9-in. spacing		"	"	23.2-38.9	35.8-78.6	0.25	68
4 3/4-in. spacing	3/16-in. holes	(23) Allerton Strom & Treybal	"	Kerosene	24.7-136	33.8-162	0.65	--
2-in. spacing	$(Re)_o$=417-1150	present data	"	Toluene	18.1-38.4	9.13-63.6	0.50	26.6
	"		Toluene	Water	38.4	13.5-68.6	1.85	10
1/16-in holes	$(Re)_o$=1300-1500		Water	Toluene	38.4-58.0	30.7-41.1	0.42	20.4

$(HTU)_T$ for the present work from Figure 20. The results are shown in Table 3.

It is seen from Table 3 that the values of $(HTU)_W$ reported by various investigators are not much different from those in this study, but they are much smaller than the $(HTU)_T$ values, so that the toluene film resistance is the controlling factor in mass transfer. The difference between $(HTU)_T$ values in the literature and this study, however, is appreciable, and the $(HTU)_T$ values from the present work are the lowest.

By comparison with the data of Treybal and Dumoulin (22), it is evident that lower values of $(HTU)_W$ and $(HTU)_T$ from the present work are due to lower plate spacing and superimposition of pulsation.

$K_W a$ values were also taken from various investigators and plotted in Figure 30 for comparison. A remarkable increase of $K_W a$ values in the present work is observed.

Holdup.—At the end of a run, it was practically impossible to predetermine the final position of the plunger after it came to rest. If the pump stopped just after its revolving arm passed the switch which opened the solenoid valve for the toluene feed, the toluene holdup would be greater. Due to this difficulty, the holdup analyses were not very accurate. The results of holdup analysis as a function of V_W, V_T, and Re are shown in Table 4 and plotted in Figures 28 and 29. The toluene holdup varied from 5 to 48%, and the water holdup from 5.3 to 5.9%.

Figure 28 shows that under constant pulsation the toluene holdup was increased by increasing the toluene rate and only slightly affected by the water rate. This result agrees with the data reported by Appel and Elgin (5), and Row, Koffolt, and Withrow (21) on packed columns, but disagrees with the data of Allerton, Strom and Treybal (23) on a perforated column.

The effect of pulsation on holdup, shown in Figure 29, was large, in both streamline and turbulent regions. As the Reynolds number increased, the size and its rising velocity of dispersed toluene in the column decreased, so the toluene holdup increased.

In transition from streamline to turbulent region, the toluene holdup suddenly dropped from 48 to 7%, shown in Figure 29. This sudden drop of toluene holdup was due to the fact that the dispersed toluene changed its form from cell-like films into drops upon transition from the streamline to the turbulent region. Since toluene drops will rise up faster in a continuous water phase than will toluene films, the amount of toluene held up in the column was less for the turbulent region than for the streamline.

With water as the discontinuous phase, the water holdup was found to be less than the toluene holdup when toluene was dispersed under the same flow rates and the same pulsation, and this is shown in Figure 28. From Run No.46 to 49, the water rate was kept constant, and the water holdup found to be constant. This fact shows that the holdup was increased by the flow rate of the discontinuous phase and is practically independent of the flow rate of the continuous phase.

Plate Efficiency.—Under constant flow rates of both phases, the individual plate efficiencies based on water phase were determined with varying pulsations. The results are shown in Table 5 and plotted in Figure 30. For the top four or five plates, the plate efficiencies remained fairly constant. From the sixth plate down to the bottom, the plate efficiencies dropped off rapidly depending on the Reynolds number.

From Figure 30 it is seen that the plate efficiencies for the top five plates varied from 16 to 32 %. Lower plate efficiencies from 1 to 17% were obtained for plates in the lower part of the column, and these low values were due to the close approach to equilibrium between the entering toluene feed and the exit water stream. The percentage of reaching the equilibrium by the exit water stream was found to be about 90 and 95% in the streamline and turbulent regions respectively.

CONCLUSIONS

1. The performance of the perforated-plate column for liquid-liquid extraction was greatly improved by superimposing pulsations. As compared with the results in literature, transfer coefficients found in this work are higher.

2. A critical Reynolds number based on the average velocity through the perforations was found to be around 1200, above which the rate of increase of the transfer coefficient was much more rapid with an increase in the Reynolds number. Pressure drop across the column increased with the Reynolds number in a similar manner.

3. Toluene film resistance was found to be the controlling factor. Water film coefficients are about 100 to 200 times larger than the toluene film coefficients.

4. Lower transfer coefficients were obtained with water as the discontinuous phase. This decrease was due to the increase of water film resistance which was more than enough to offset the decrease of toluene film resistance.

5. High flow rate ratio of water totoluene gave abnormally low $(HTU)_{ow}$ values, which could not be correlated by plotting $(HTU)_{ow}$ versus $\frac{HV_w}{V_T}$.

6. A modified HTU method of correlation is recommended, and general equations including the effect of pulsation are presented.

7. The holdup increases with increasing flow rate of the discontinuous phase, and independent of flow rate of the continuous phase.

With constant flow rates, holdup increases exponentially with an increase in the Reynolds number.

8. Individual plate efficiencies were found to be rather low, ranging from 1 to 35 per cent.

NOMENCLATURE

a	=	interfacial area, sq.ft.per cu.ft.of effective column volume
A	=	contact area, sq.ft.
C	=	Concentration of benzoic acid, lb.mols.per cu.ft.of solution
C_1, C_2	=	constants
dC_w/dC_T	=	slope of equilibrium distribution curve
ΔC	=	concentration difference, lb.mols.per cu.ft.solution
$(\Delta C)_m$	=	log.mean of concentration difference driving forces at the extremities of the column, lb.mols.per cu.ft.solution
D	=	diameter of the perforations on the plate, ft.
E	=	plate efficiency, per cent
F	=	force, dynes
g	=	acceleration due to gravity, cm/sec.^{2}
G	=	mass velocity, lbs./hr.ft.^{2}
H	=	Henry's constant, equal to dC_w/dC_T
HTU	=	height of transfer unit, ft.
$(HTU)_{ow}$	=	height of an overall transfer unit based on the water phase, ft.
HTU_w	=	height of water film transfer unit, ft.
HTU_T	=	height of toluene film transfer unit, ft.
k	=	film transfer coefficient, lb.mols.benzoic acid transferred /hr.sq.ft. $(\Delta C)_m$
$K_w a$	=	overall transfer coefficient based on water phase, lb. mols.benzoic acid transferred /hr.cu.ft. $(\Delta C)_m$
$k_w a$	=	water film transfer coefficient, lb.mols.benzoic acid transferred /hr.cu.ft. $(\Delta C)_m$
$k_T a$	=	toluene film transfer coefficient, lb.mols.benzoic acid transferred /hr.cu.ft. $(\Delta C)_m$

L = length, ft.

M = mass, pound

n' = number of transfer units

N = number of lb.mols.of benzoic acid transferred

Re = Reynolds number through the perforations on the plate, equal to $DV\rho / \mu$

Sc = Schmidt number

V = velocity of the mixture of toluene and water through the perforations on the plate, ft.per sec.

V' = effective volume of the column, cu.ft.

V_T = flow rate of toluene, cu.ft./hr.sq.ft.

V_w = flow rate of water, cu.ft./hr.sq.ft.

V_σ = volume of a drop, cc.

W_c = flow rate of continuous phase, lb./hr.sq.ft.

W_d = flow rate of discontinuous phase, lb./hr.sq.ft.

$d, e, f, l, m, n, r, s, t$ = exponents

θ = time, hrs.

σ = surface tension, dynes per cm.

μ = viscosity of the toluene-water mixture, lb./sec.ft.

ρ = density of the toluene-water mixture, lb./cu.ft.

$\Delta\rho$ = difference in density between dispersed and continuous phases

$\phi', \phi'', \phi_T, \phi''_T, \phi'_W, \phi''_W$ = constants

Subscripts

B	=	buoyant	R	=	resistance
C	=	continuous phase	T	=	toluene
D	=	discontinuous phase	W	=	water
K	=	kinetic	σ	=	surface tension
o	=	perforation			
O	=	overall			

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BIBLIOGRAPHY

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APPENDIX

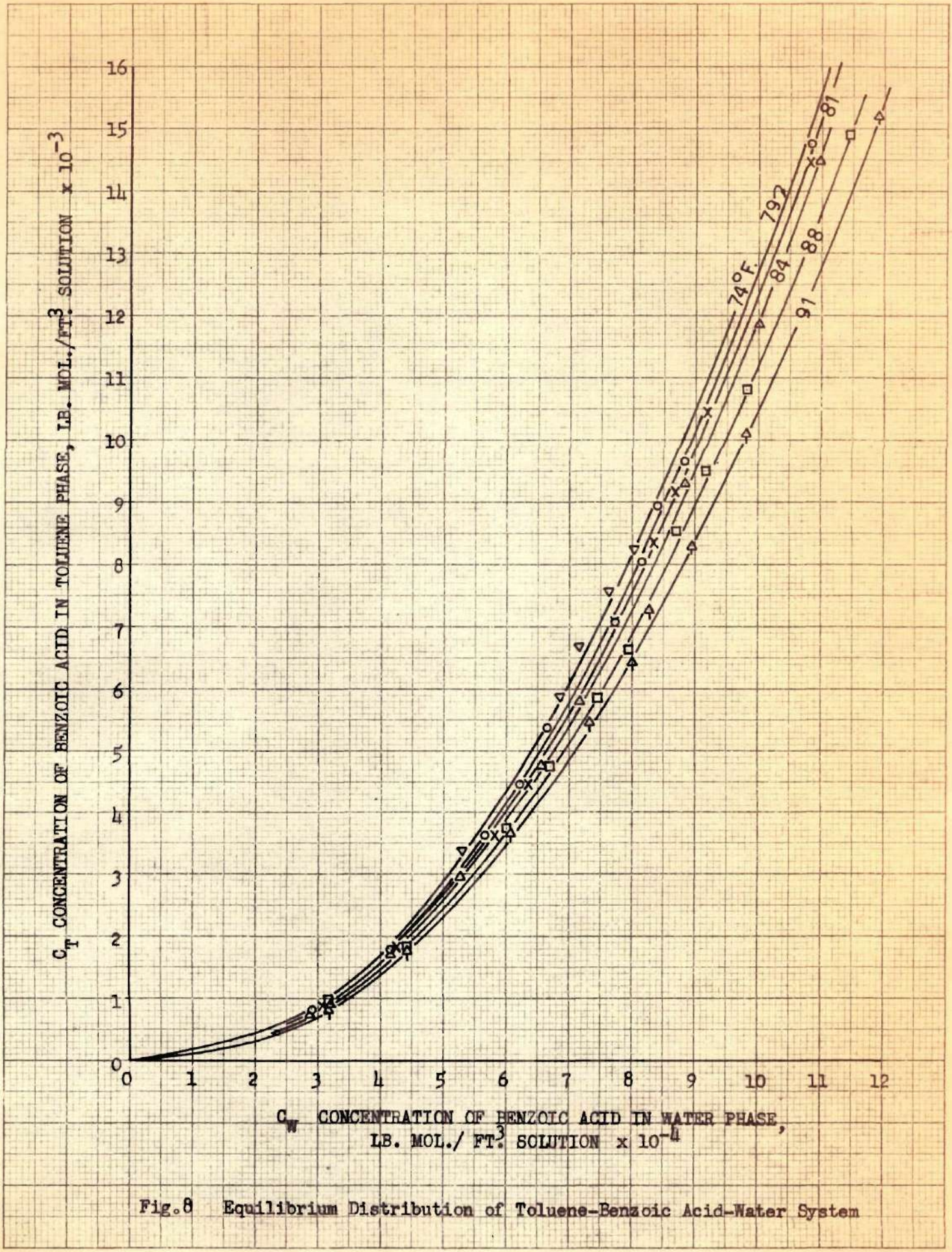


Fig.8 Equilibrium Distribution of Toluene-Benzoic Acid-Water System

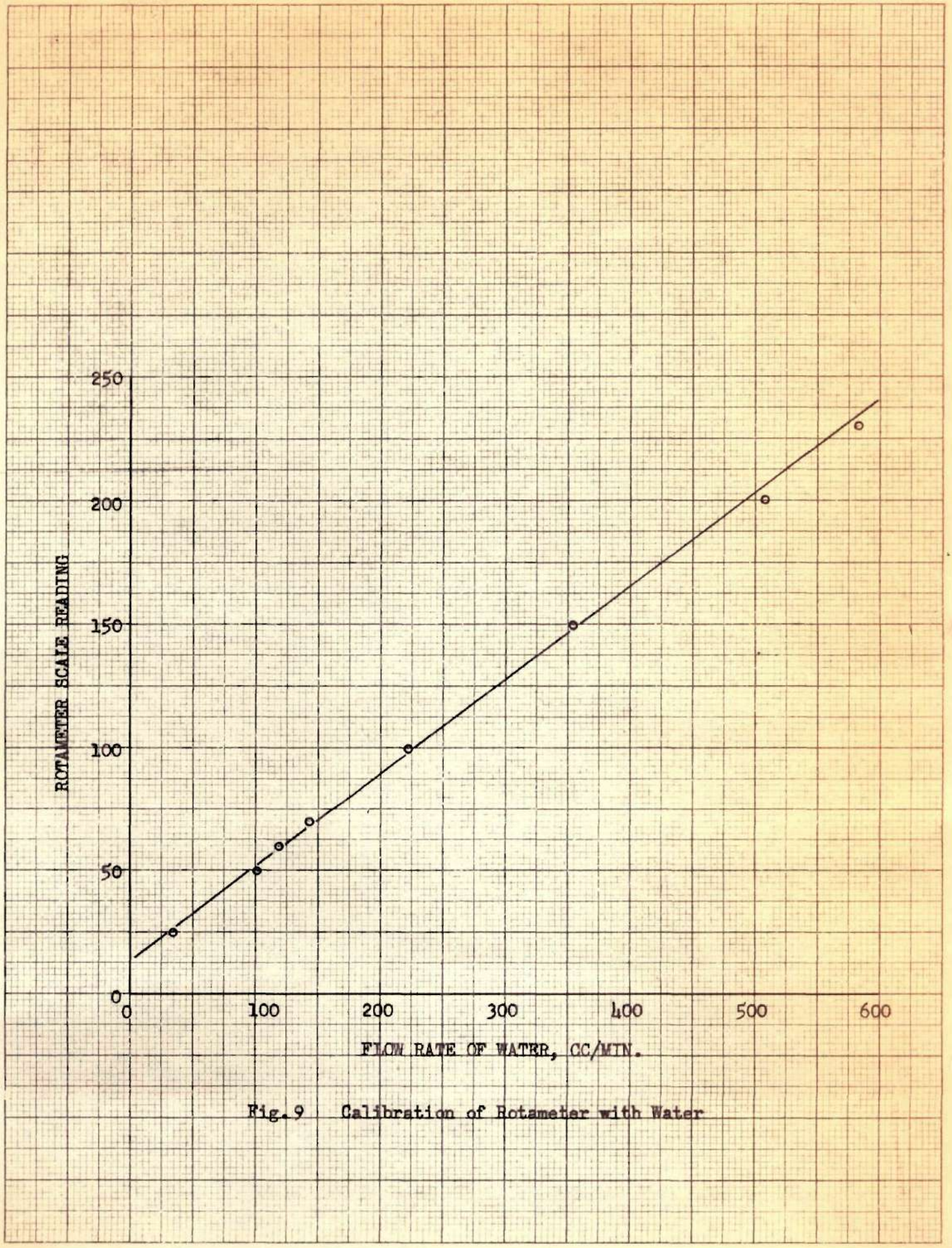


Fig. 9 Calibration of Rotameter with Water

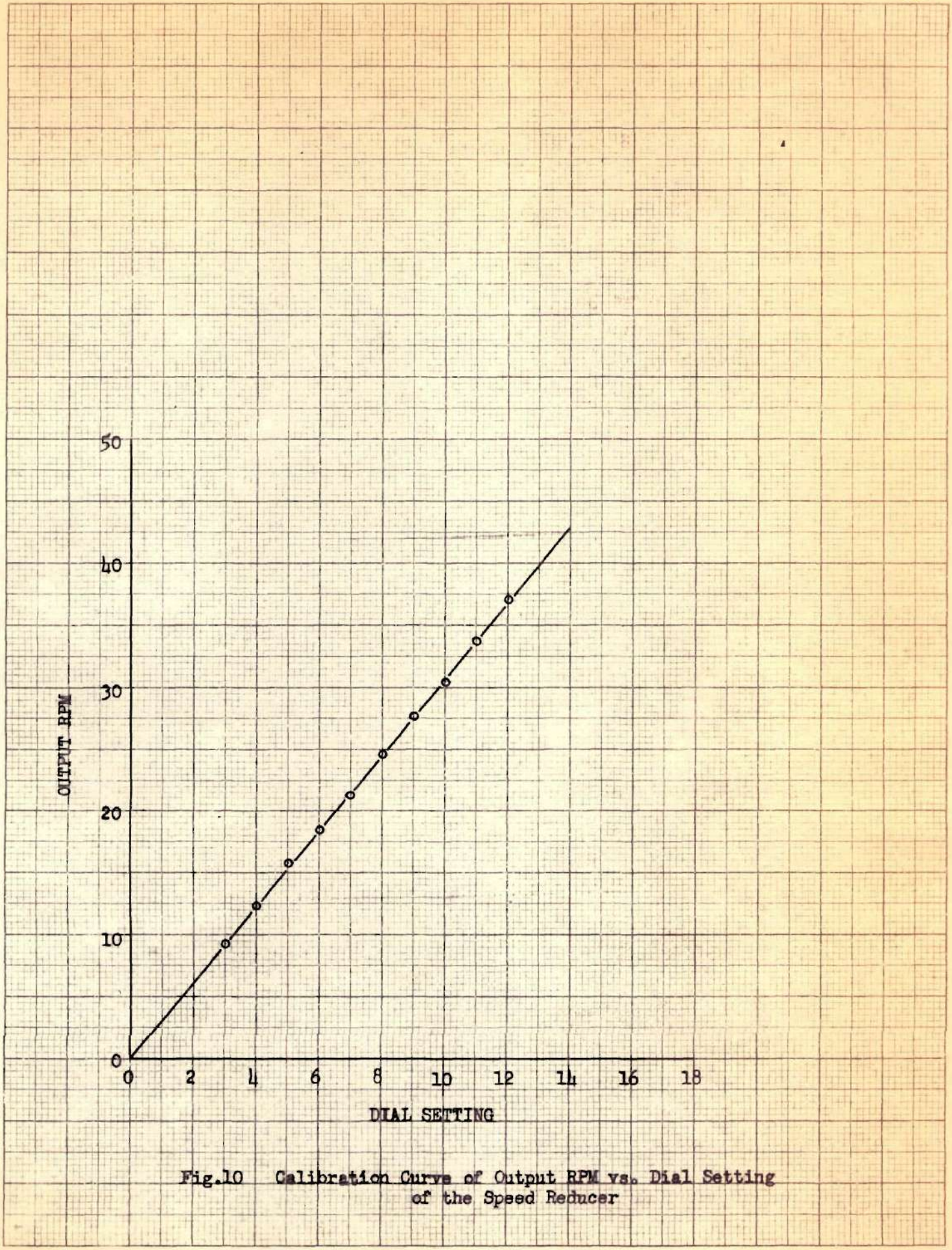


Fig.10 Calibration Curve of Output RPM vs. Dial Setting of the Speed Reducer

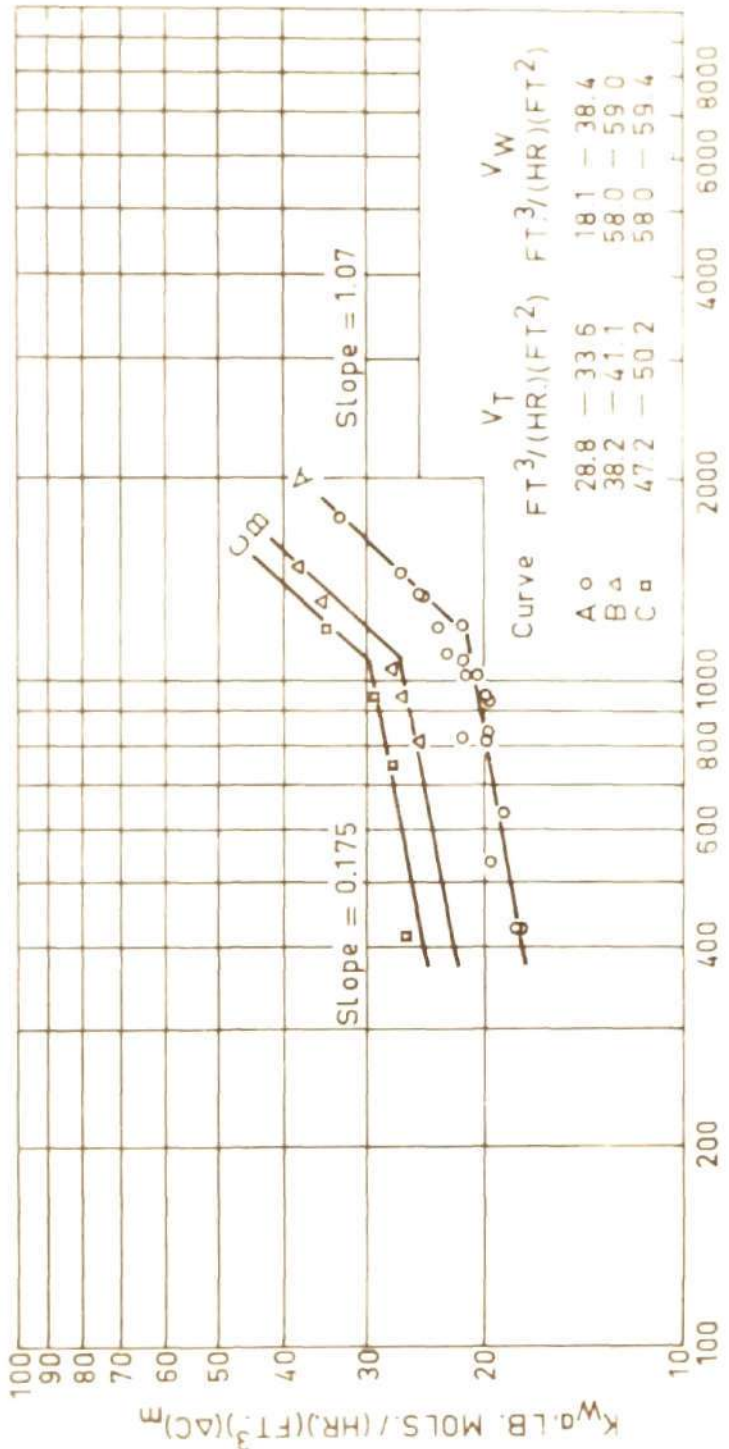


Fig 11 Effect of Pulsation on Overall Transfer Coefficient

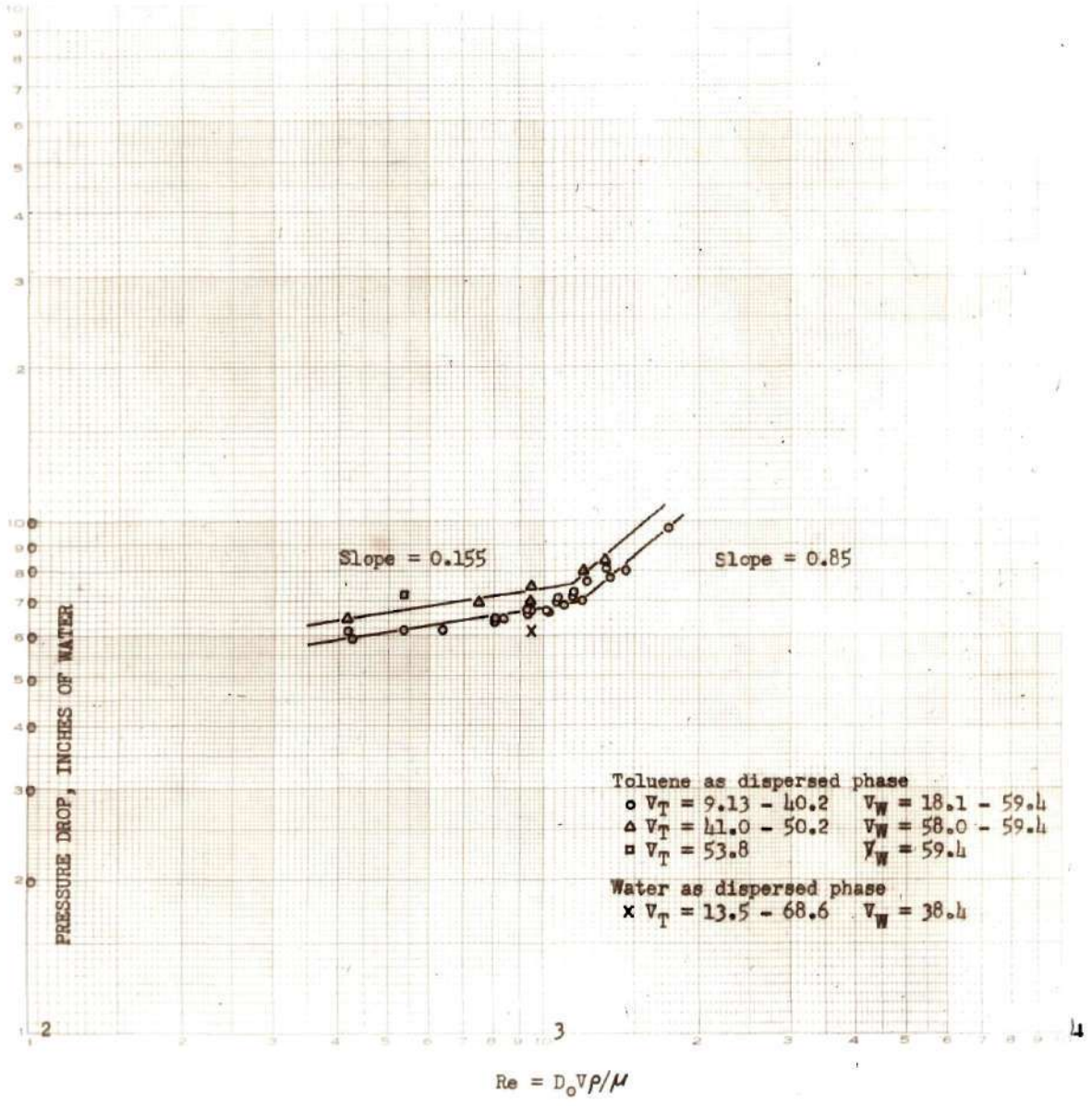


Fig.15 Effect of Pulsation on Pressure Drop

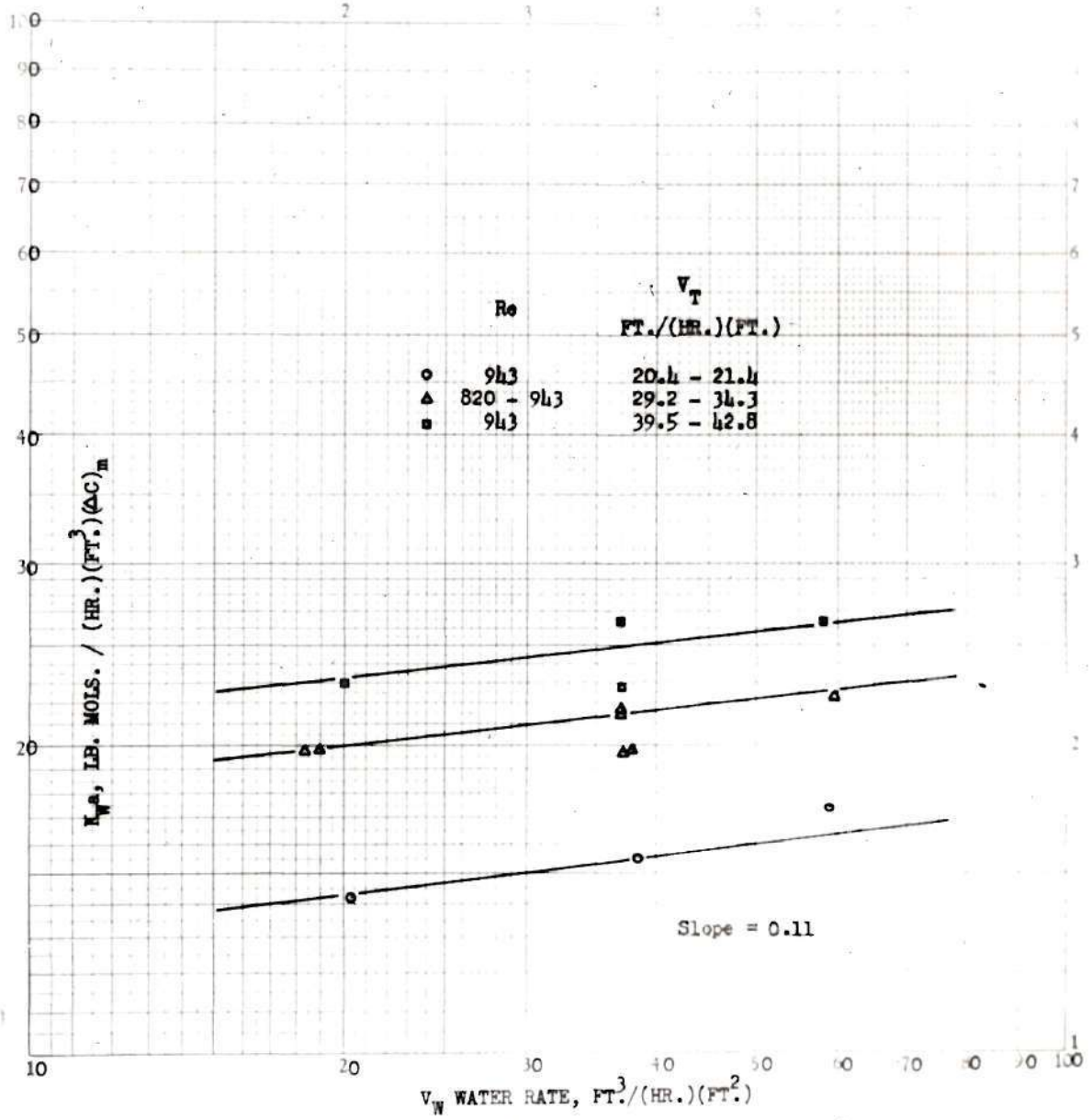


Fig. 16 The Effect of Water Rate on Overall Transfer Coefficient in the Streamline Region

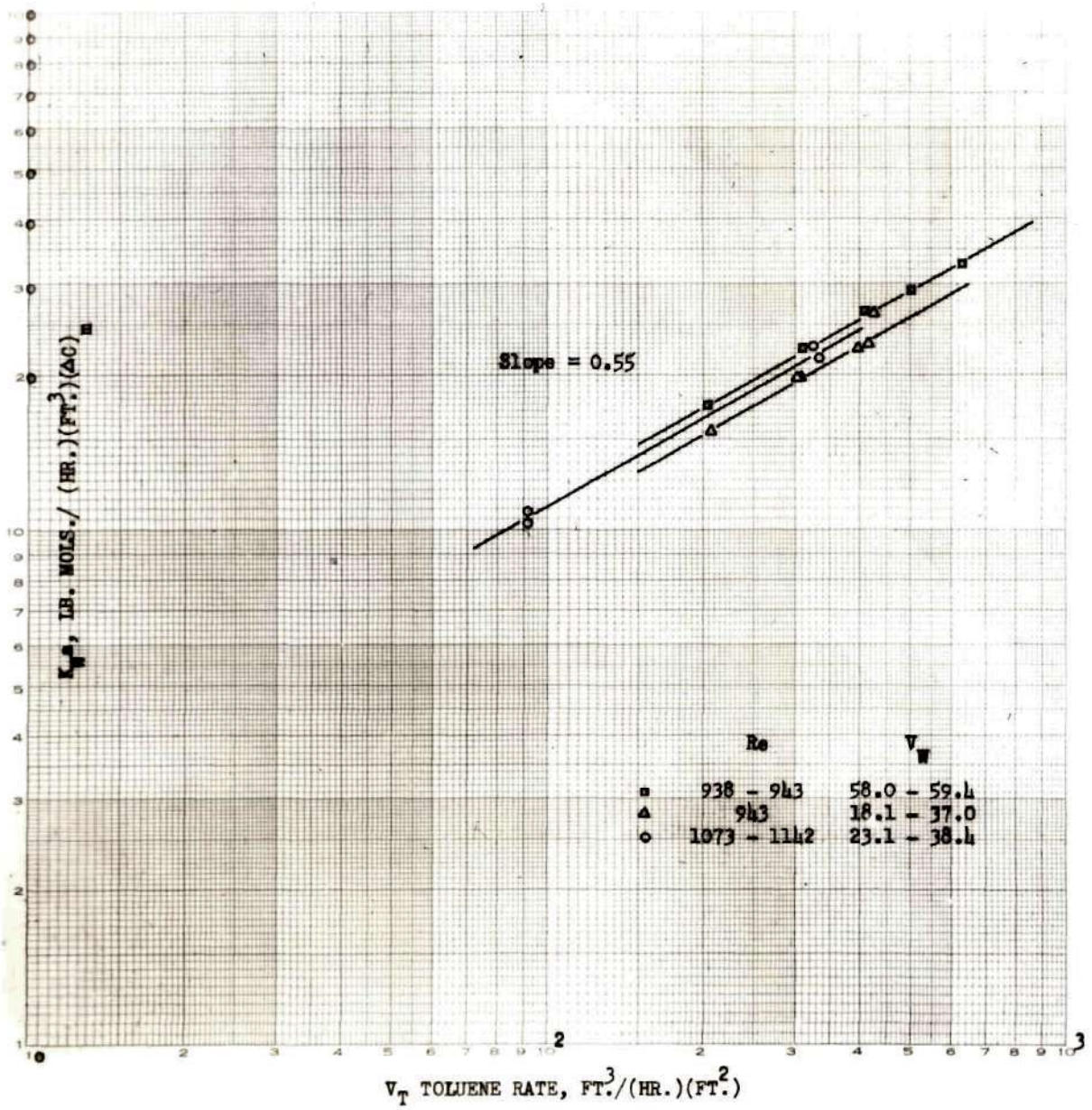


Fig. 17 Effect of Toluene Rate on Overall Transfer Coefficient in Streamline Region

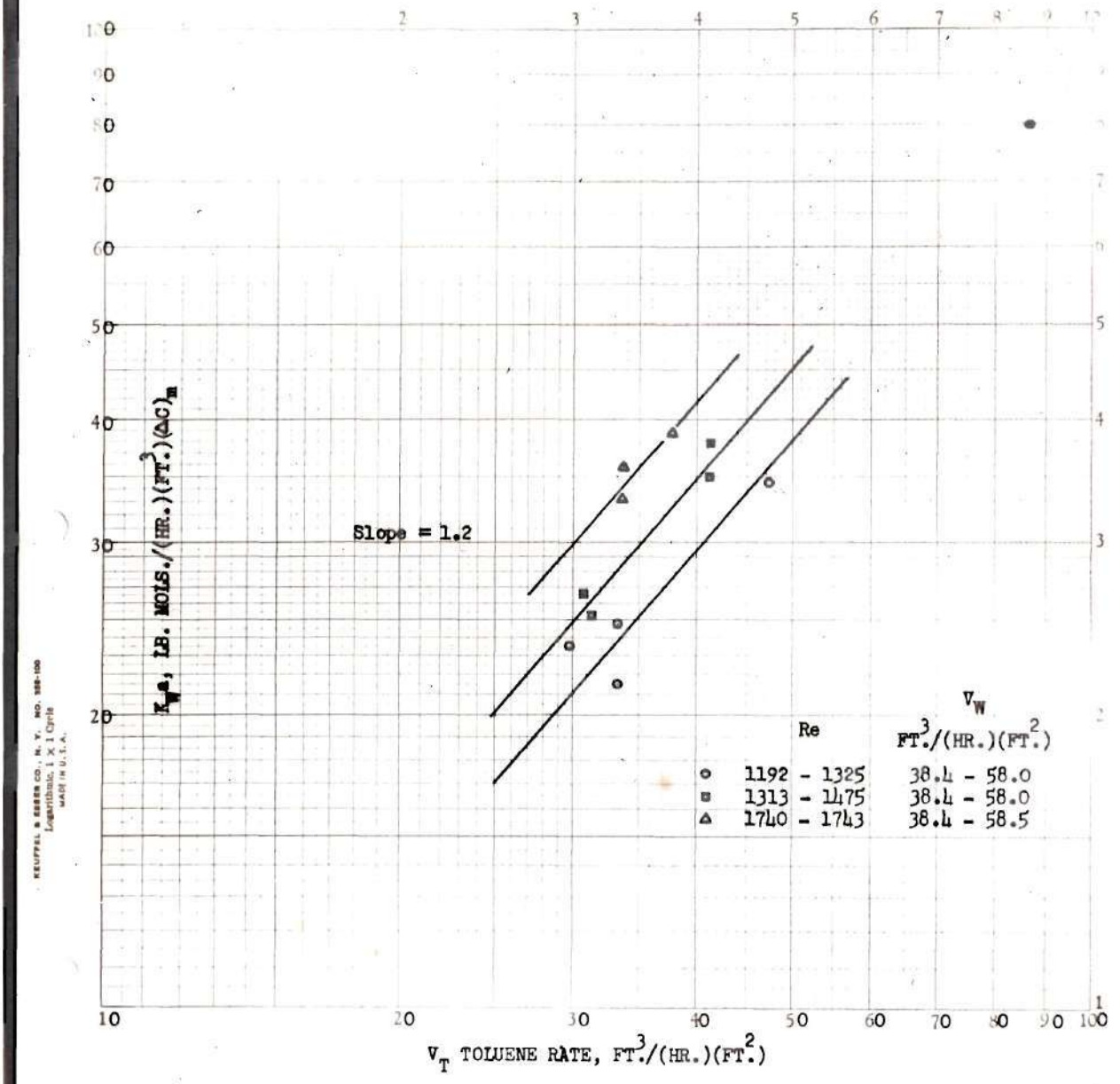


Fig. 18 The Effect of Toluene Rate on Overall Transfer Coefficient in the Turbulent Region

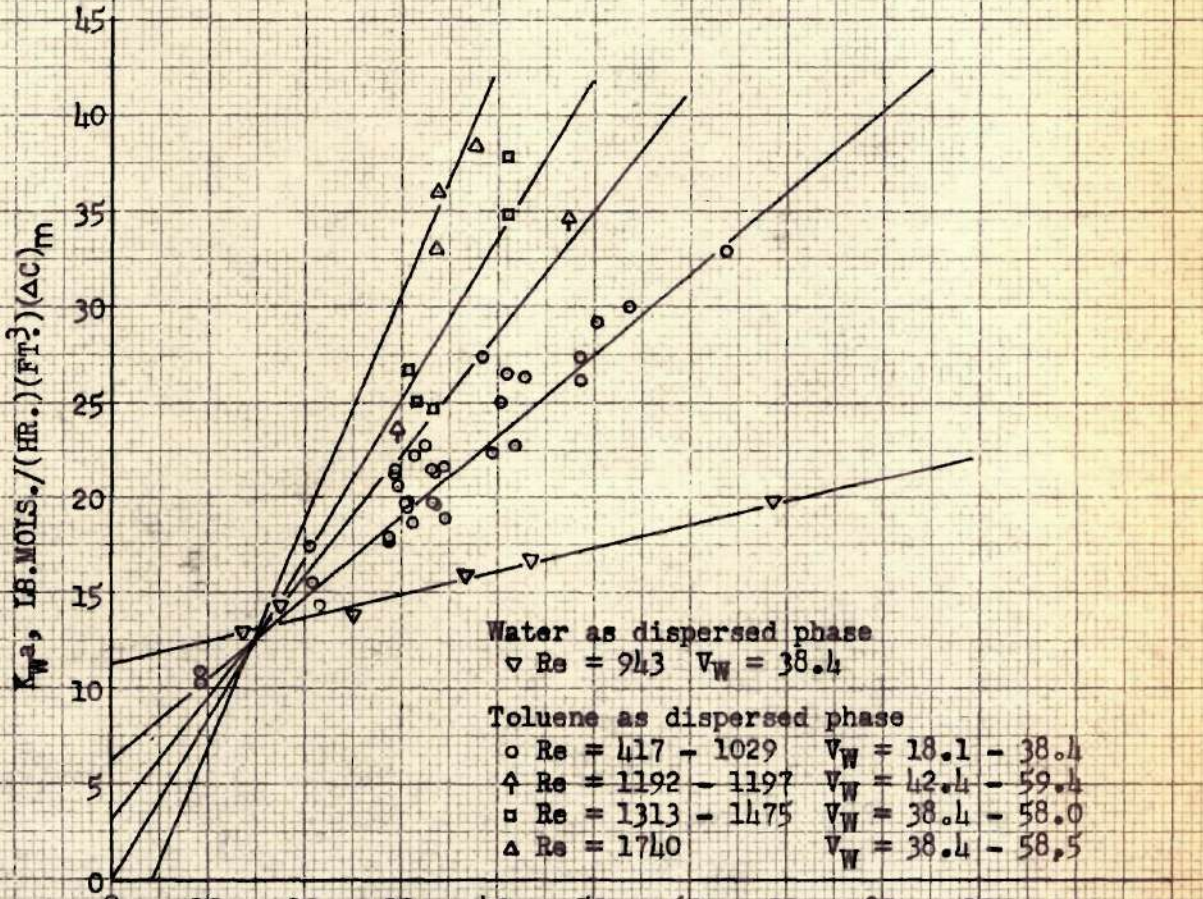


Fig.19 Overall Extraction Coefficient for the Pulse Column

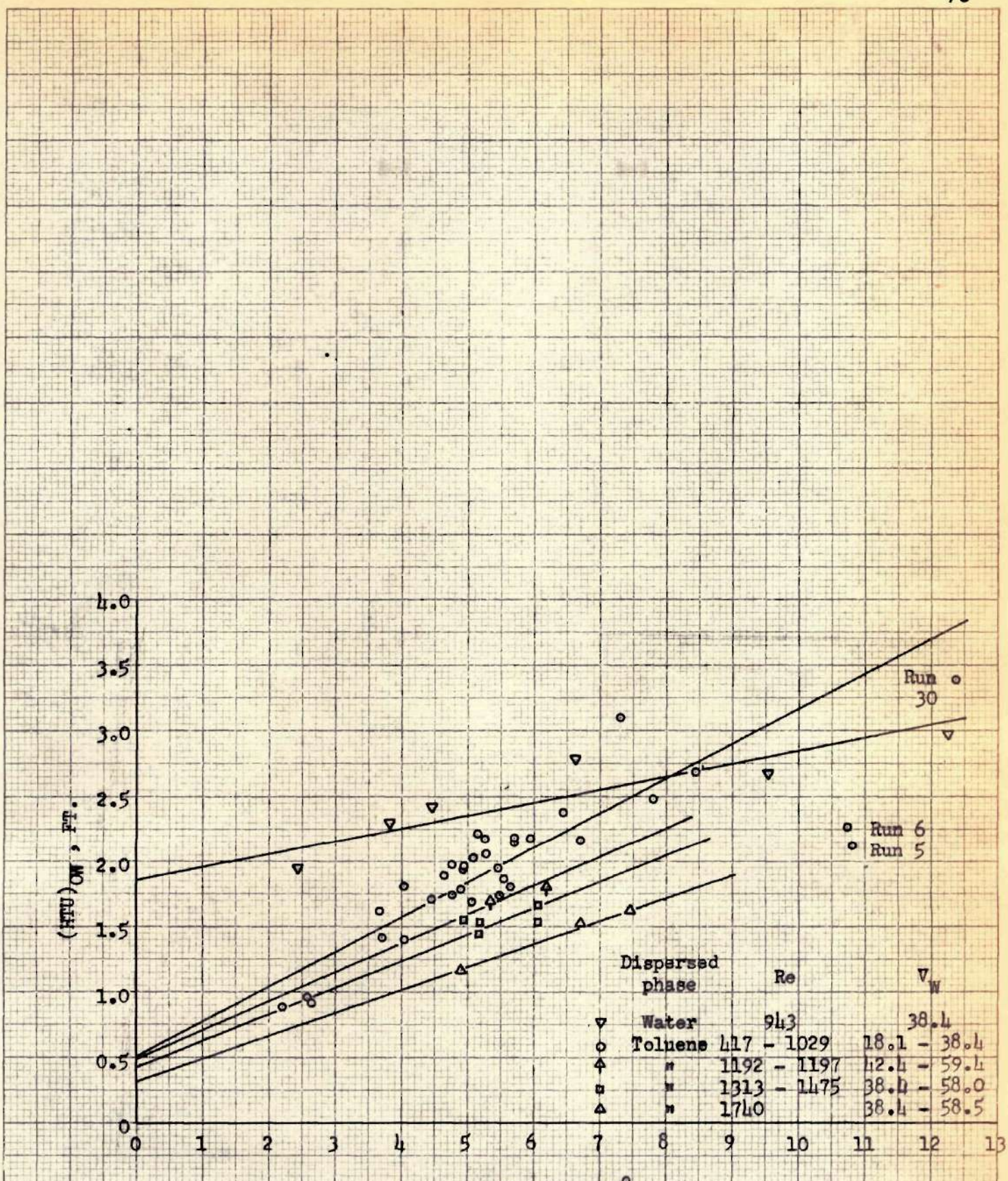


Fig.20 Effect of Pulsation on Overall $(HTU)_{OW}$

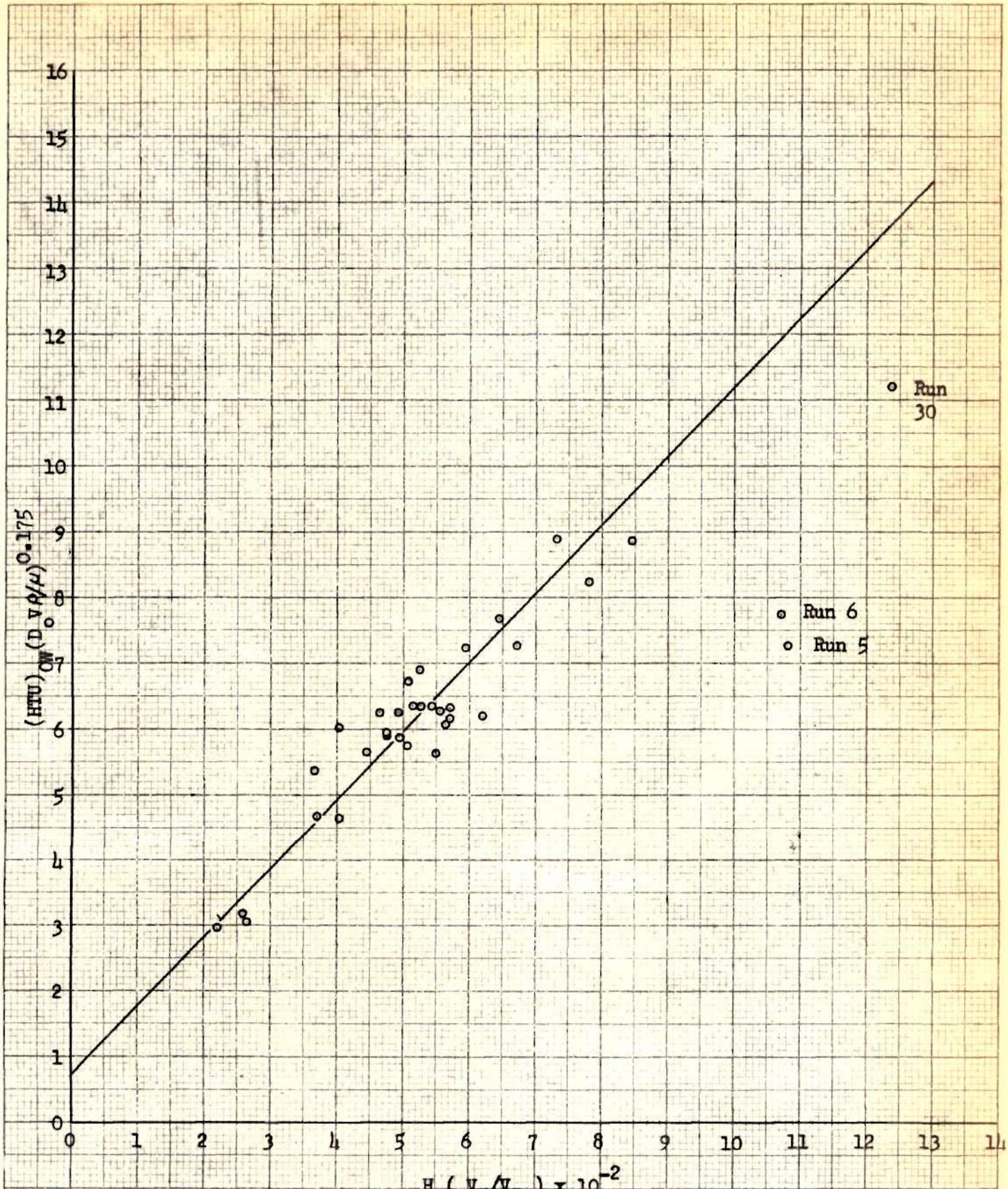


Fig. 21 Overall $(HTU)_{OW}(Re)^{0.175}$ vs. $H(V_W/V_T)$ Plot in the Streamline Region

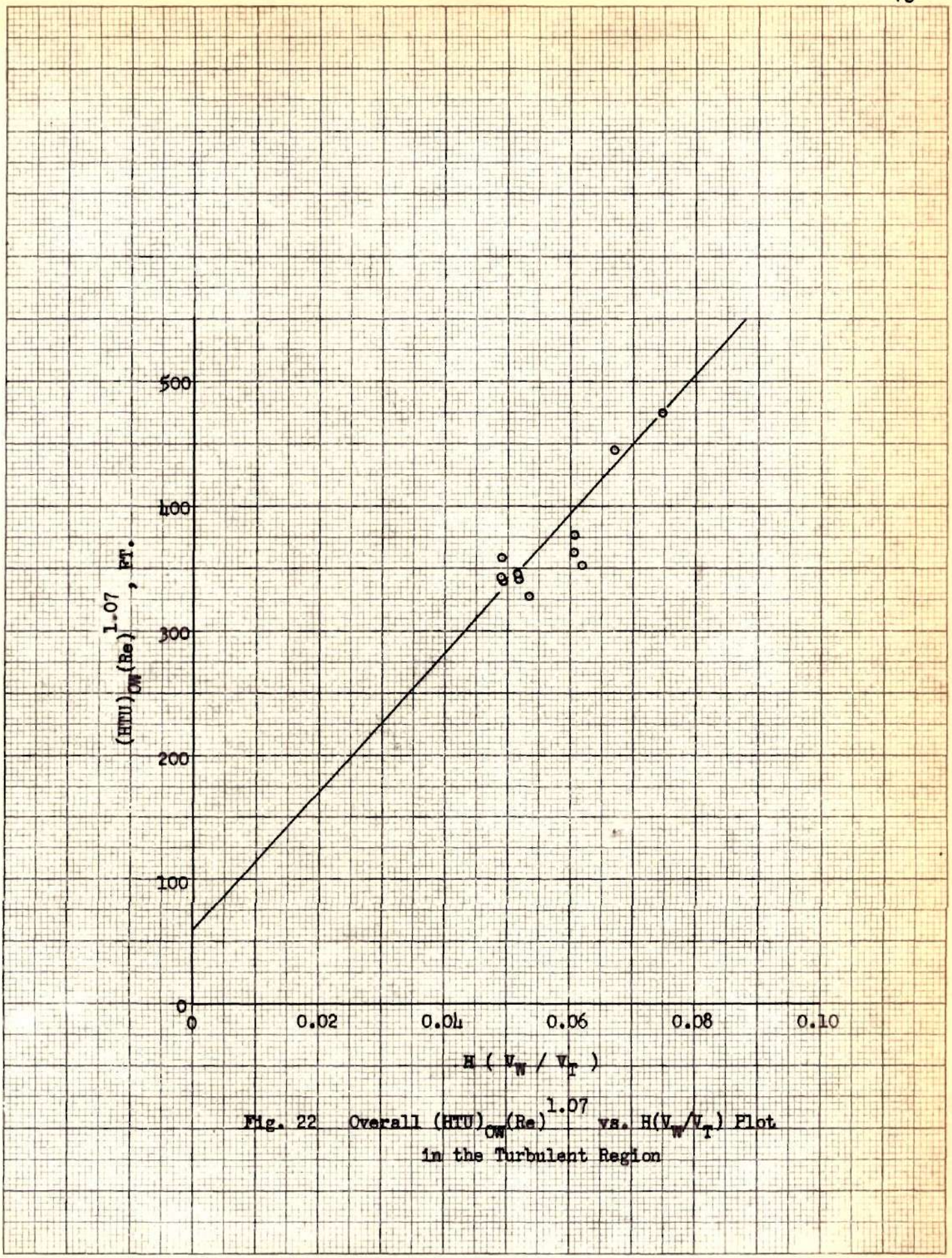


Fig. 22 Overall $(HTU)_{OW}(Re)^{1.07}$ vs. $H(V_W/V_T)$ Plot in the Turbulent Region

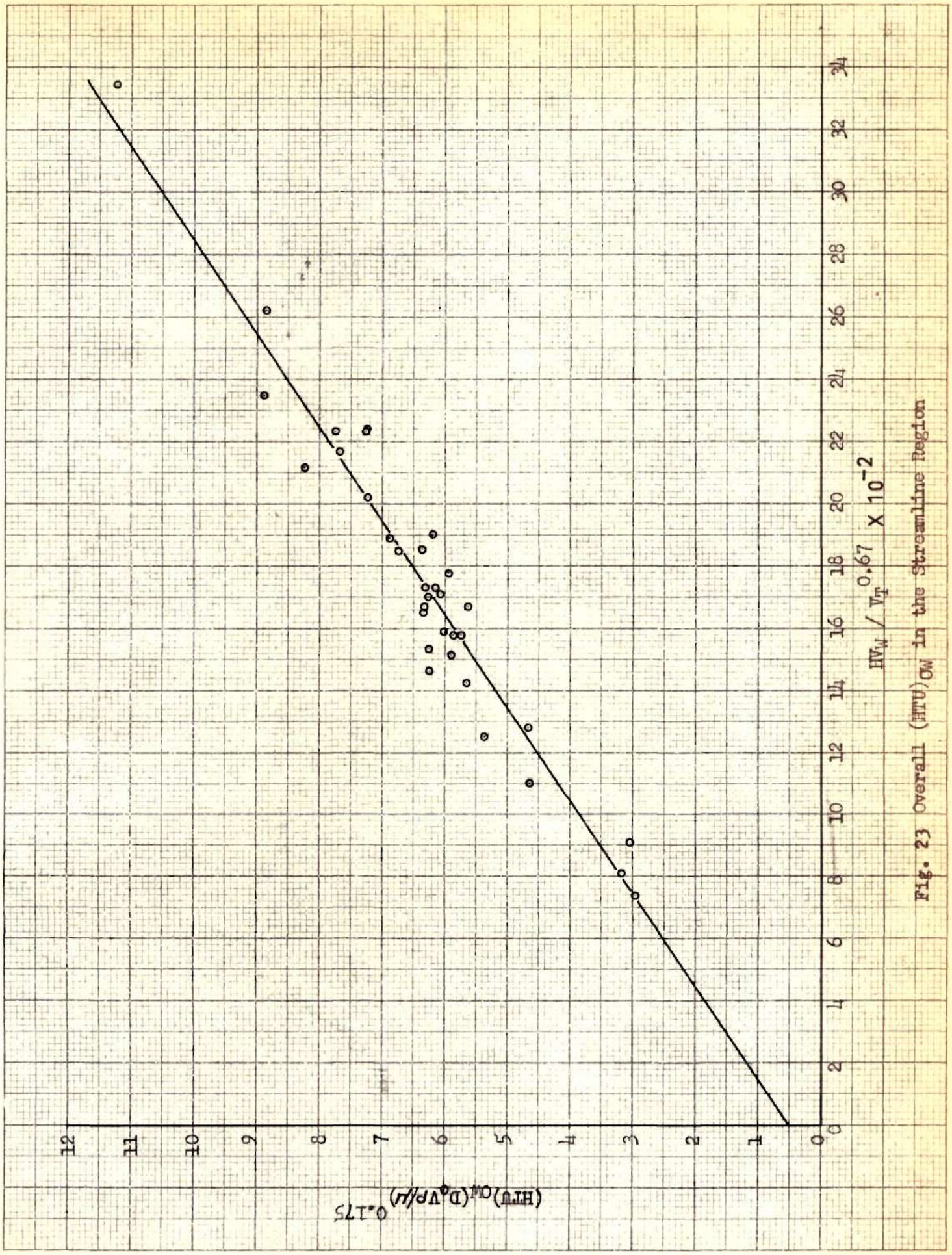


Fig. 23 Overall $(HTU)_{OW}$ in the Streamline Region

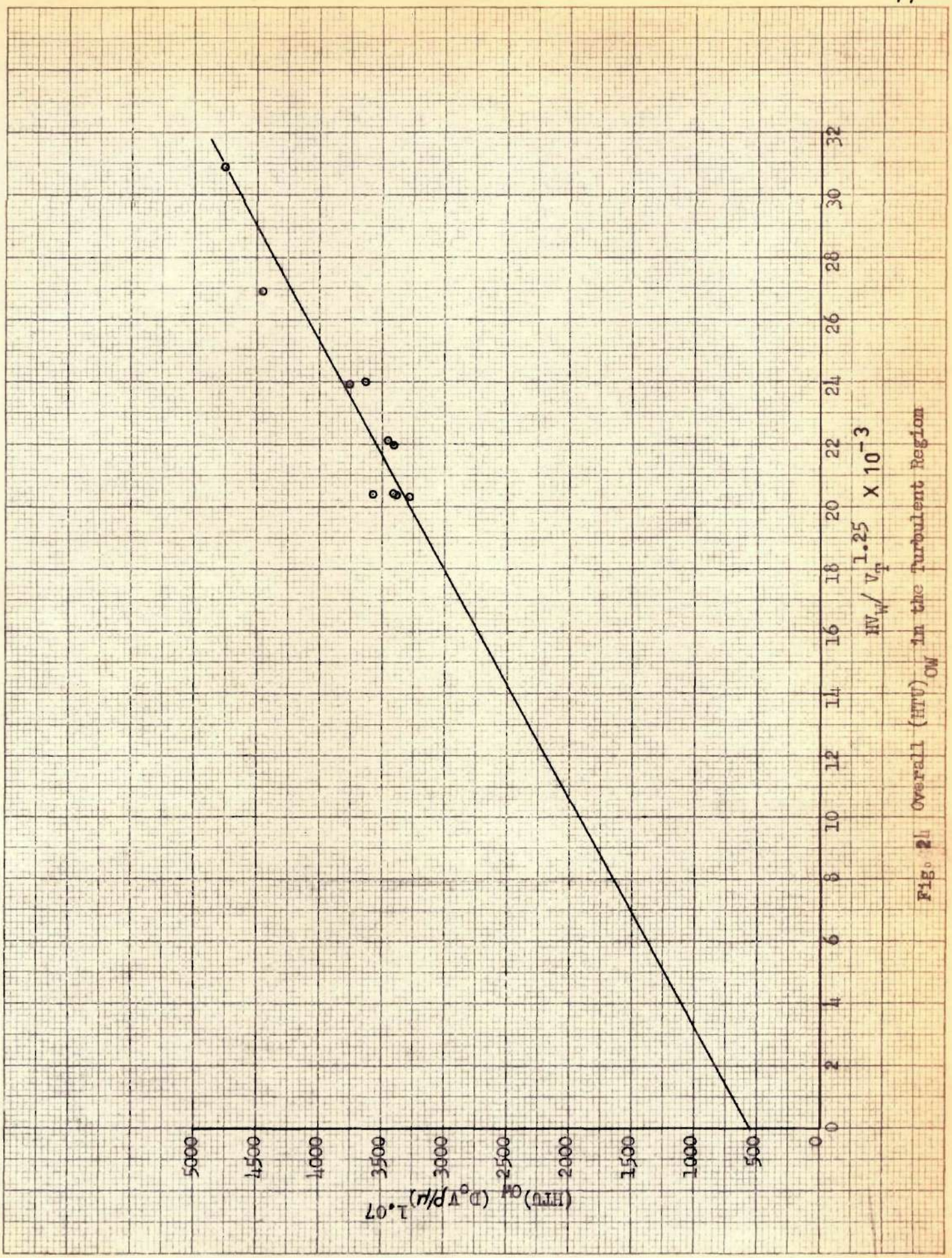


Fig. 21 Overall $(HTU)_{OW}$ in the Turbulent Region

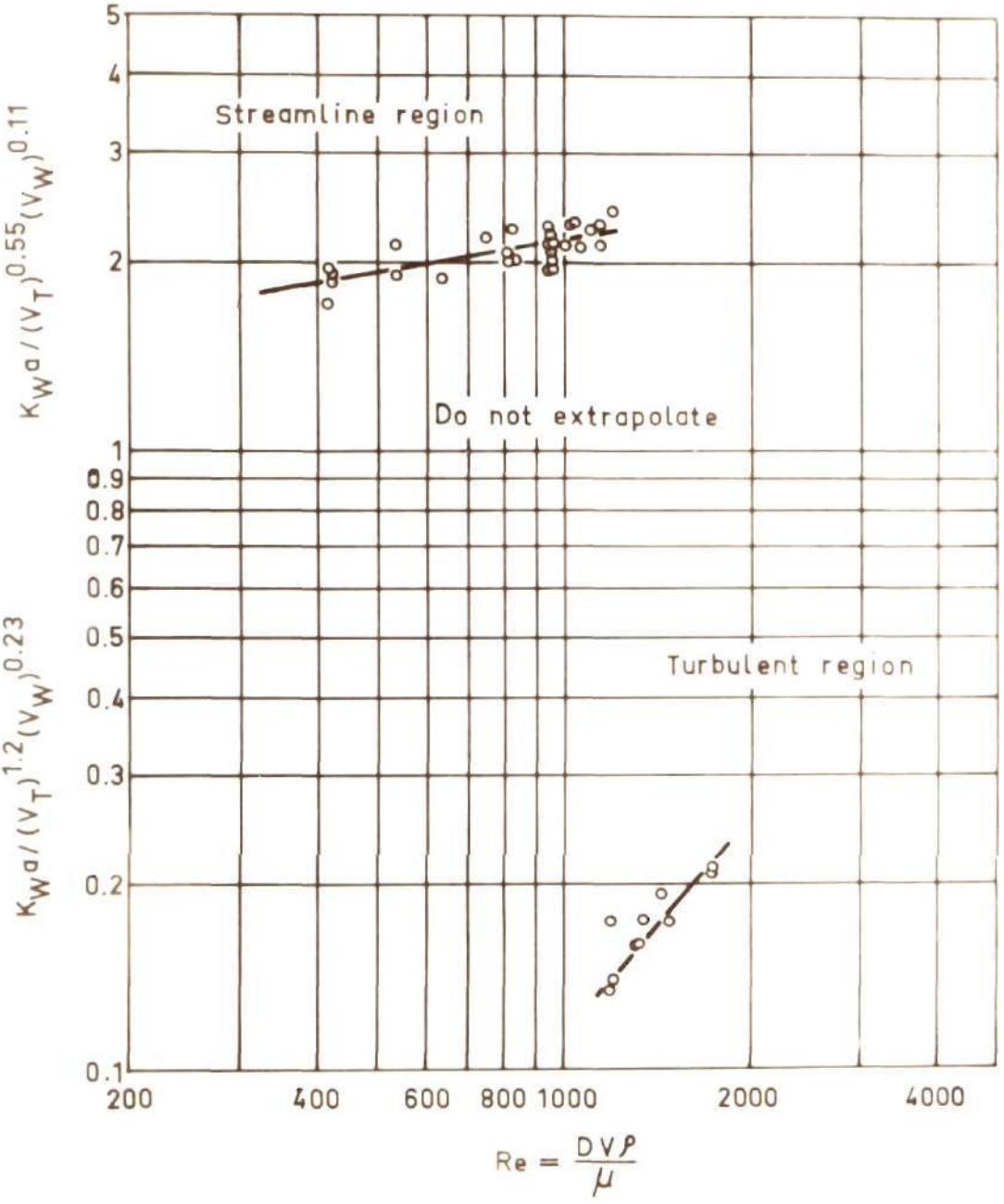


Fig. 25. Empirical Correlation of Overall Transfer Coefficient with Reynolds Number

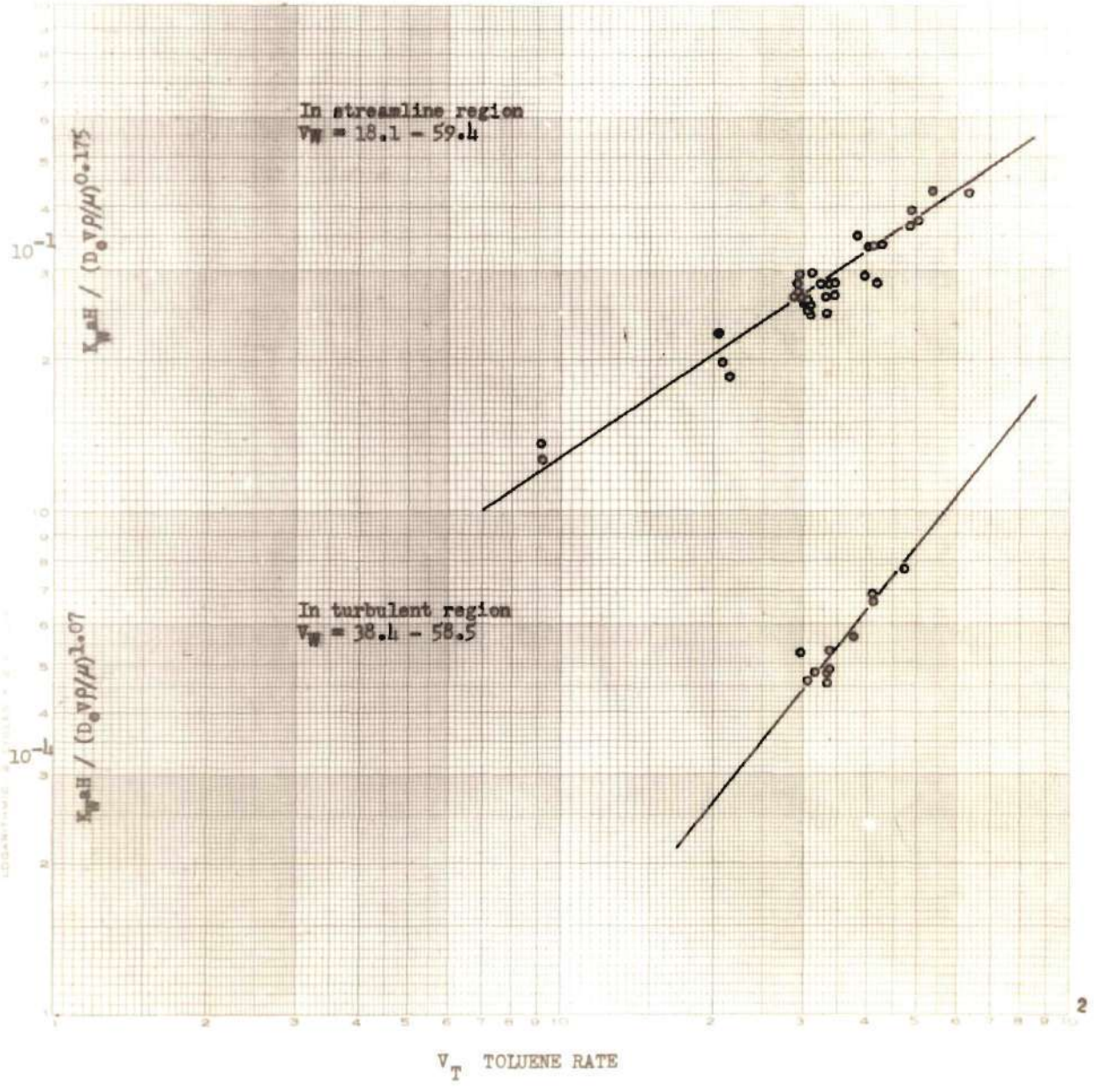


Fig.26 Effect of Toluene Rate on $K_W a$ for the Pulse Column

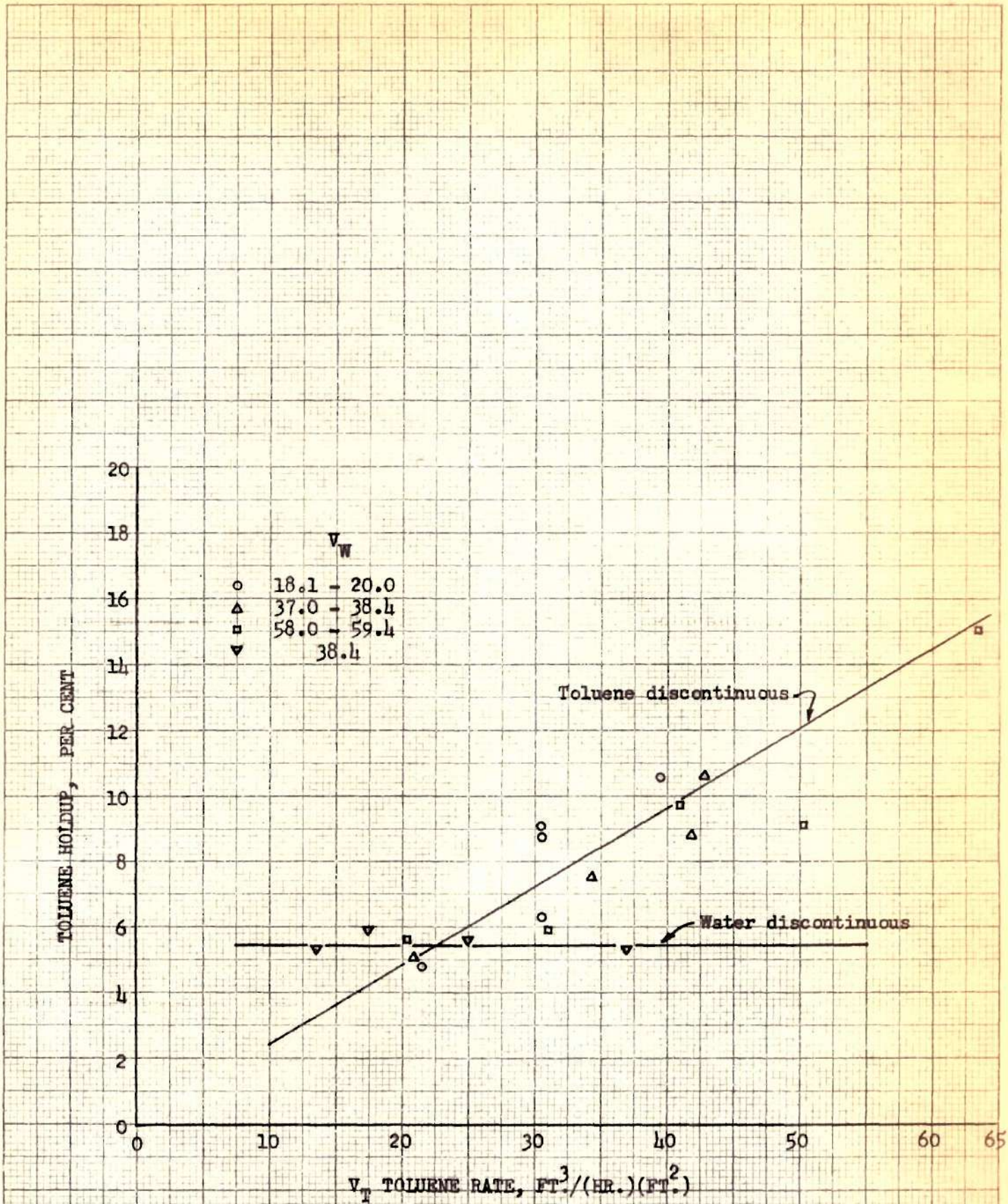


Fig. 28 Effect of Toluene Rate on Toluene Holdup
Re = 943

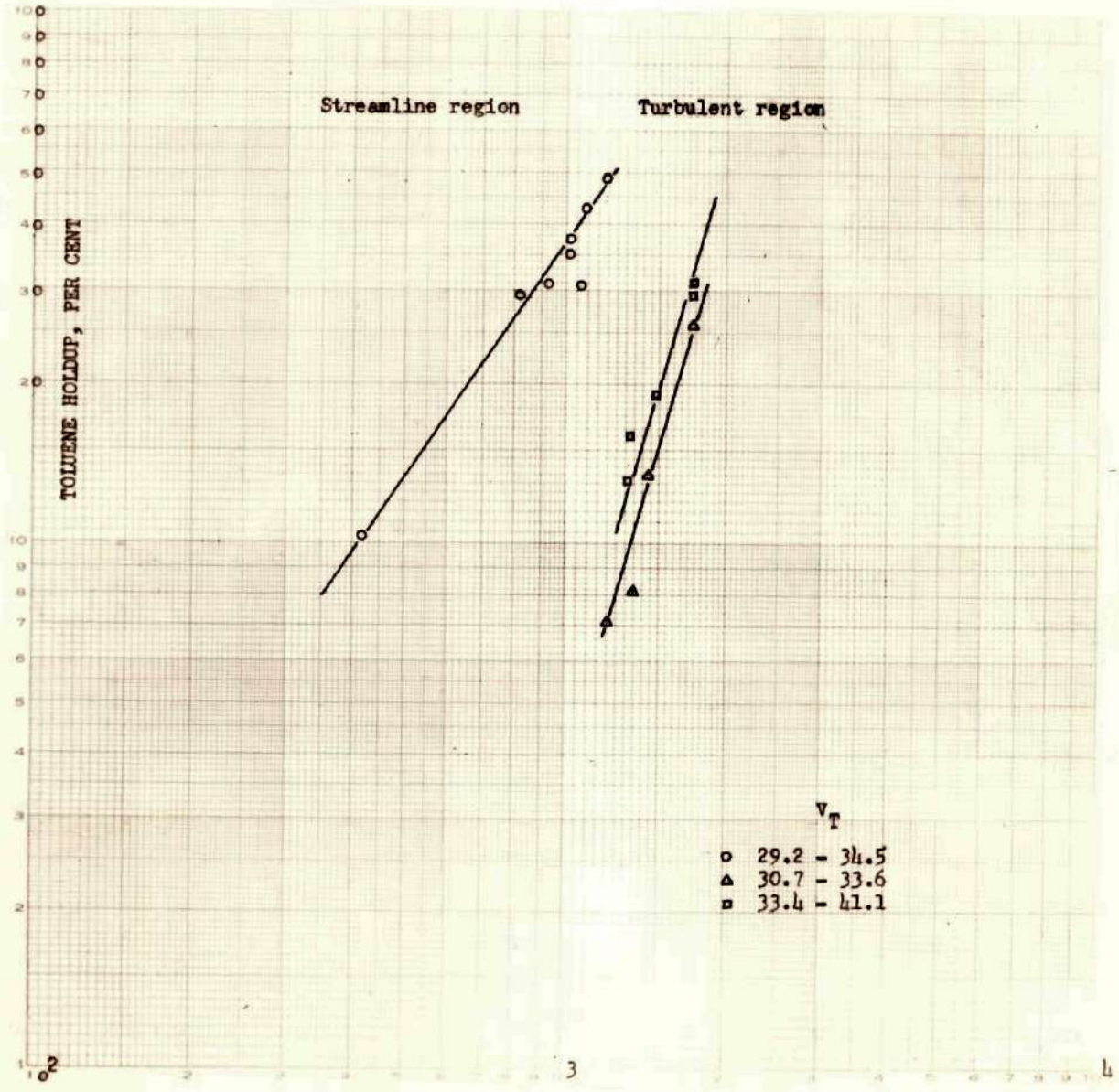
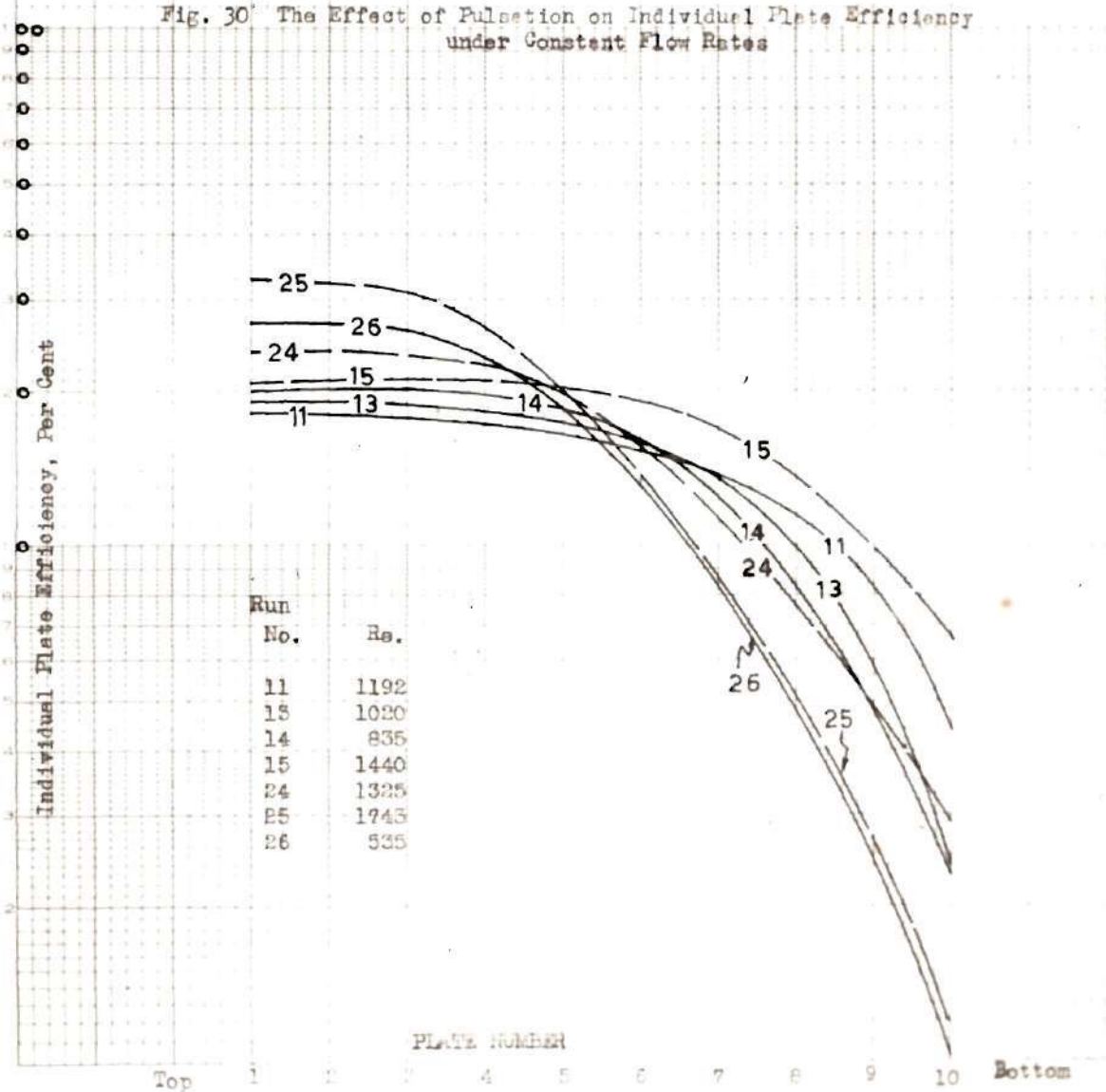


Fig. 29 Effect of Pulsation on Toluene Holdup

Fig. 30 The Effect of Pulsation on Individual Plate Efficiency under Constant Flow Rates



Curve	Reference	Plate spacing inches	Hole dia. inch	V_W $FT.^3/HR.FT^2$	Re
A	Allerton, Strom, and Treybal	4.75	3/16	37.5 - 77	---
B	Treybal and Dumoulin	3	3/16	16.4	---
C	"	6	"	23.2 - 40.4	---
D	"	9	"	23.5 - 38.9	---
E	Row, Koffolt, and Withrow	6	1/8	11.5 - 37.0	---
F		6	3/12	11.8 - 37.1	---
G	Present data	2	1/16	18.1 - 38.4	417 - 1150
H	"	2	1/16	38.4 - 58.0	1300 - 1500

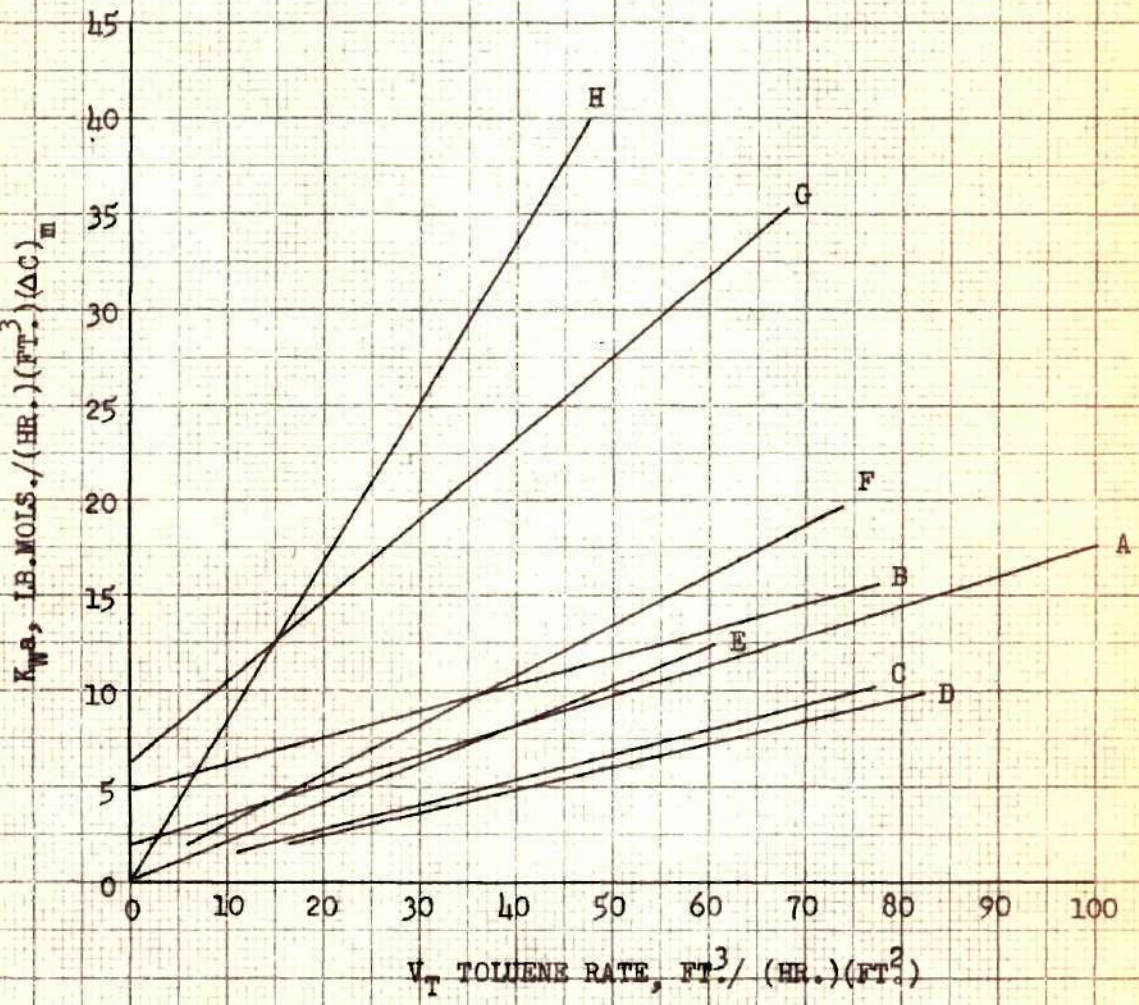


Fig. 31. Comparison of K_Wa Values from the Literature

Table 4. Summary of Data and Results.

Run No.	Temp. °F.	Flow Rates		Concentrations lb.mol/ft.^{3}		
		Water ft.^{3}/hr.ft.^{2}	Toluene ft.^{3}/hr.ft.^{2}	Toluene In	Toluene Out	Water Out
1	83	20.0	21.4	0.01135	0.01042	0.000992
2	93	18.1	30.4	0.01144	0.01084	0.001041
3	76	19.0	30.6	0.01131	0.01075	0.000935
4	86	20.0	39.5	0.01138	0.01088	0.000989
5	80	23.1	9.13	0.01110	0.00902	0.000835
6	79	23.1	9.16	0.01137	0.00931	0.000828
7	77	38.4	20.8	0.01211	0.01060	0.000820
8	82	38.4	28.8	0.01093	0.00983	0.000844
9	82	38.4	28.8	0.01061	0.00952	0.000824
10	81	37.4	29.2	0.01118	0.01007	0.000895
11	86	42.4	29.6	0.01133	0.01005	0.000917
12	82	38.4	29.4	0.01064	0.00948	0.000858
13	82	38.4	29.7	0.01140	0.01024	0.000895
14	81	38.4	30.3	0.01124	0.01018	0.000878
15	75	38.4	30.7	0.01142	0.01028	0.000898
16	81	38.4	31.1	0.01135	0.01029	0.000865
17	69	38.4	31.1	0.01167	0.01065	0.000832
18	79	38.4	31.5	0.01130	0.01022	0.000913
19	78	38.4	32.3	0.01150	0.01046	0.000898
20	77	37.5	33.1	0.01145	0.01048	0.000887
21*	80	38.4	32.7	0.01120	0.01025	0.000835
22	74	37.2	33.3	0.01154	0.01060	0.000865
23	79	38.4	33.4	0.01130	0.01028	0.000873
24	82	38.4	33.4	0.01125	0.01020	0.000917
25	83	38.4	33.6	0.01127	0.01013	0.000943
26	86	38.4	33.6	0.01160	0.01057	0.000899
27	75	37.0	34.3	0.01138	0.01047	0.000875
28	75	37.0	41.8	0.01137	0.01062	0.000882
29	81	37.0	42.8	0.01113	0.01035	0.000923
30	82	58.9	20.4	0.01133	0.00933	0.000720
31	92	59.4	31.1	0.01130	0.00973	0.000849
32	84	58.5	33.8	0.01133	0.00978	0.000920
33	82	58.5	34.5	0.01138	0.01012	0.000750
34	85	58.5	37.8	0.01126	0.00987	0.000935
35	85	59.4	38.2	0.01128	0.00998	0.000867
36	86	59.4	40.2	0.01136	0.01012	0.000845
37	83	58.0	41.0	0.01118	0.00994	0.000912
38	77	58.0	41.0	0.01139	0.01025	0.000840
39	82	58.0	41.1	0.01123	0.00995	0.000917
40	86	58.0	41.2	0.01125	0.01014	0.000927
41	84	58.0	48.7	0.01129	0.01030	0.000860
42	83	59.4	50.2	0.01130	0.01027	0.000870
43	82	59.4	48.5	0.01133	0.01025	0.000834
44	84	59.4	53.8	0.01143	0.01046	0.000892
45	83	59.4	63.6	0.01138	0.01053	0.000898

Table 4 Continued.

Run No.	Temp. °F.	Flow Rates		Concentrations lb.mol/ft.^{3}		
		Water ft.^{3}/hr.ft.^{2}	Toluene ft.^{3}/hr.ft.^{2}	Toluene In	Toluene Out	Water Out
46#	84	38.4	13.5	0.01132	0.00922	0.000768
47#	84	38.4	17.4	0.01120	0.00946	0.000785
48#	83	38.4	25.0	0.01102	0.00984	0.000778
49#	83	38.4	36.9	0.01127	0.01040	0.000821
50#	83	38.4	43.3	0.01108	0.01033	0.000838
51#	85	38.4	68.6	0.01122	0.01071	0.000884

* Discarded.

Water as the dispersed phase.

Table 4 Continued.

Pressure Drop in.water	Hold-up per cent	Pulsation Stroke length inches	Pulsation Frequency RPM	Re $\frac{DV\rho}{\mu}$	Henry's constant H	$K_w a$ lb.mol. $\frac{}{(hr.)(ft.^3)(\Delta C)_m}$
67.7	4.8	1.44	21.7	943	0.0430	14.3
66.5	9.1	1.44	21.7	943	0.0443	19.8
70.5	8.8	1.44	21.7	943	0.0418	19.8
68.6	10.6	1.44	21.7	943	0.0434	22.4
71.0	--	1.88	20.2	1142	0.0427	10.9
73.0	--	1.88	20.2	1142	0.0426	10.2
68.0	5.0	1.44	21.7	943	0.0421	15.5
59.0	--	1.44	9.75	425	0.0429	17.5
59.0	--	1.44	9.75	425	0.0429	17.9
63.6	29.4	1.44	18.9	820	0.0428	21.5
70.0	48.5	1.44	27.4	1192	0.0434	23.5
66.5	37.2	2.72	12.4	1020	0.0429	21.9
67.0	35.0	2.72	12.4	1020	0.0429	20.5
64.5	--	2.25	12.3	835	0.0428	19.7
80.0	13.5	2.25	20.2	1440	0.0414	26.7
61.5	--	2.25	9.35	635	0.0428	18.6
64.0	--	1.44	18.5	804	0.0400	19.8
77.8	8.1	1.75	25.5	1347	0.0426	25.1
68.7	42.8	1.44	25.1	1090	0.0424	22.8
71.1	30.3	1.44	24.7	1073	0.0421	21.5
70.0	--	2.25	15.7	1070	0.0427	17.0
67.5	30.9	1.44	21.3	926	0.0413	19.7
76.4	7.1	1.44	27.7	1205	0.0426	21.5
81.0	16.0	1.44	30.4	1325	0.0429	24.7
97.0	25.6	1.44	40.1	1743	0.0430	33.0
61.5	--	.64	27.7	535	0.0433	19.6
68.6	7.5	1.44	21.7	943	0.0412	21.7
70.6	8.8	1.44	21.7	943	0.0412	22.8
67.0	10.6	1.44	21.7	943	0.0428	26.3
68.0	5.6	1.44	21.7	943	0.0429	17.4
69.1	5.9	1.44	21.6	938	0.0441	22.2
--	29.4	1.44	40.1	1740	0.0431	36.0
61.0	10.3	1.44	9.6	417	0.0429	18.9
--	31.0	1.44	40.1	1740	0.0432	38.4
66.5	--	2.72	12.5	1029	0.0432	27.4
64.1	--	1.44	18.5	805	0.0434	25.0
84.0	13.1	1.75	24.8	1313	0.0430	34.9
68.5	9.7	1.44	21.7	943	0.0421	26.5
--	19.1	2.25	21.7	1475	0.0429	37.9
80.0	12.8	1.44	27.5	1197	0.0434	34.5
64.5	--	1.44	9.6	417	0.0431	26.2
75.0	9.1	1.44	21.7	944	0.0430	29.2
69.5	--	1.75	14.2	750	0.0429	27.4
72.0	--	0.64	27.7	535	0.0431	30.0
--	5.0	1.44	21.7	943	0.0430	32.9

Table 4 Continued.

Pressure Drop in.water	Hold-up per cent	Pulsation Stroke length inches	Pulsation Frequency RPM	Re $\frac{D V \rho}{\mu}$	Henry's constant H	$K_w a$ $\frac{\text{lb.mol.}}{(\text{hr})(\text{ft}^3)(\Delta C)_m}$
61.5	5.3	1.44	21.7	943	0.0431	12.9
61.0	5.9	1.44	21.7	943	0.0431	14.3
61.8	5.6	1.44	21.7	943	0.0430	13.8
61.3	5.3	1.44	21.7	943	0.0430	15.9
63.0	--	1.44	21.7	943	0.0430	16.7
65.3	--	1.44	21.7	943	0.0432	19.8

Table 4 Continued.

HTU_{ow} ft.	$H(\frac{V_w}{V_T})$	$(HTU)_{ow}(Re)^{l}$ $l = 0.175$ $l = 1.07$	$\frac{HV_w}{(V_T)^{0.67}}$	$\frac{HV_w}{(V_T)^{1.25}}$	$\frac{k_w a}{(V_w)^{0.11}(V_T)^{0.55}}$
1.40	0.0402	4.65	0.1101	----	1.905
0.914	0.0264	3.04	0.0905	----	2.203
0.960	0.0259	3.19	0.0804	----	2.194
0.892	0.0220	2.96	0.0739	----	2.128
2.12	0.1080	7.27	0.2240	----	2.285
2.26	0.1073	7.75	0.2230	----	2.133
2.48	0.0780	8.24	0.2118	----	1.955
2.19	0.0570	6.31	0.1733	----	1.845
2.14	0.0570	6.16	0.1733	----	1.895
1.74	0.0548	5.64	0.1670	----	2.255
1.80	0.0620	35.10	0.1900	0.02665	2.41
1.81	0.0562	6.08	0.1710	----	2.282
1.87	0.0553	6.28	0.1700	----	2.120
1.95	0.0544	6.34	0.1673	----	2.020
1.44	0.0517	34.58	----	0.0221	---
2.06	0.0527	6.36	0.1645	----	1.880
1.94	0.0492	6.27	0.1536	----	2.000
1.53	0.0520	34.12	----	0.02195	---
1.69	0.0505	5.75	0.1581	----	2.250
1.74	0.0476	5.90	0.1514	----	2.100
2.26	0.0500	7.66	0.1585	----	1.673
1.89	0.0463	6.25	0.1465	----	1.918
1.79	0.0490	35.80	----	0.0204	---
1.55	0.0493	34.00	----	0.0204	---
1.16	0.0490	34.10	----	0.0204	---
1.96	0.0494	5.88	0.1582	----	1.892
1.71	0.0445	5.66	0.1425	----	2.085
1.62	0.0365	5.38	0.1250	----	1.971
1.41	0.0370	4.60	0.1279	----	2.242
3.38	0.1236	11.22	0.3345	----	2.089
2.68	0.0843	8.87	0.2620	----	2.101
1.62	0.0746	47.50	----	0.03085	---
3.10	0.0730	8.90	0.2346	----	1.699
1.52	0.0670	44.52	----	0.0269	---
2.16	0.0670	7.28	0.2232	----	2.318
2.38	0.0642	7.69	0.2165	----	2.060
1.66	0.0606	36.40	----	0.0240	---
2.18	0.0594	7.24	0.202	----	2.164
1.53	0.0605	37.61	----	0.02393	---
1.68	0.0534	32.90	----	0.02031	---
2.22	0.0514	6.37	0.1853	----	1.952
2.03	0.0508	6.74	0.185	----	2.116
2.17	0.0523	6.90	0.1889	----	2.024
1.98	0.0475	5.94	0.178	----	2.022
1.81	0.0401	6.01	0.1585	----	1.947

Table 4 Continued.

HTU_{ow} ft.	$H(\frac{V_w}{V_T})$	$(HTU)_{ow}(Re)^{l}$ $l=0.175$ $l=1.07$	$\frac{HV_w}{(V_T)^{0.67}}$	$\frac{HV_w}{(V_T)^{1.25}}$	$\frac{K_w a}{(V_w)^{0.11}(V_T)^{0.55}}$
2.98	0.1225	9.89	0.2895	---	2.062
2.68	0.0953	8.90	0.244	---	1.986
2.78	0.0662	9.23	0.191	---	1.571
2.42	0.0447	8.04	0.1474	---	1.466
2.30	0.0381	7.64	0.132	---	1.404
1.94	0.0242	6.44	0.0976	---	1.192

Table 4 Continued.

$\frac{K_w a}{V_w^{0.23}(V_T)^{1.2}}$	$\frac{K_w aH}{(Re)^l}$	$\frac{K_w a}{V_w^{0.23}(V_T)^{1.2}}$	$\frac{K_w aH}{(Re)^l}$
---	0.1852	---	0.1675
---	0.2640	---	0.1858
---	0.2492	---	0.1787
---	0.2930	---	0.206
---	0.1358	---	0.2162
---	0.1270	---	0.2579
---	0.1967		
---	0.2606		
---	0.2665		
---	0.284		
0.1725	0.296		
---	0.2707		
---	0.262		
---	0.2595		
0.1909	0.000461		
---	0.2577		
---	0.245		
0.1731	0.000480		
---	0.284		
---	0.267		
---	0.214		
---	0.246		
0.138	0.000458		
0.158	0.000482		
0.210	0.000483		
---	0.283		
---	0.269		
---	0.283		
---	0.339		
---	0.225		
---	0.296		
0.2075	0.000530		
---	0.2825		
0.193	0.000566		
---	0.351		
---	0.336		
0.1592	0.000685		
---	0.336		
0.1722	0.000661		
0.1316	0.000764		
---	0.394		
---	0.378		
---	0.369		
---	0.431		
---	0.426		

Table 5. Data on Individual Plate Concentrations and Efficiencies.

Plate concentration C_w = lb.mol.per cu.ft.solution

Plate efficiencies E = per cent

Plate number starts from top of the column

Plate No.	Run No.9		Run No.10		Run No.11	
	C_w x10-4	E	C_w x10-4	E	C_w x10-4	E
1	4.20	24.6	6.18	19.6	7.08	18.0
2	5.42	24.4	6.82	19.9	7.55	17.5
3	6.35	22.2	7.35	20.4	7.93	17.7
4	7.00	19.5	7.79	20.1	8.25	16.4
5	7.45	18.6	8.14	19.9	8.50	16.4
6	7.80	14.1	8.42	20.0	8.71	18.5
7	8.02	11.9	8.65	15.1	8.90	13.3
8	8.18	8.4	8.79	12.5	9.02	11.3
9	9.28	5.5	8.89	7.1	9.11	8.3
10	8.34	1.9	8.94	1.5	9.17	4.5

Plate No.	Run No.13		Run No.14		Run No.15	
	C_w x10-4	E	C_w x10-4	E	C_w x10-4	E
1	6.53	19.2	5.66	18.2	6.95	19.8
2	7.12	19.8	6.35	20.6	7.41	20.6
3	7.62	18.7	7.00	20.8	7.80	21.7
4	8.00	18.0	7.53	23.1	8.13	22.3
5	8.30	17.1	8.00	18.9	8.40	18.9
6	8.54	15.3	8.30	16.2	8.58	17.9
7	8.72	11.0	8.51	12.7	8.72	15.4
8	8.83	10.0	8.65	7.3	8.82	14.3
9	8.92	3.7	8.72	4.4	8.90	11.4
10	8.95	2.5	8.78	2.4	8.95	6.8

Table 5. Continued.

Plate No.	Run No.16		Run No.18		Run No.20	
	C_w x10-4	E	C_w x10-4	E	C_w x10-4	E
1	4.70	16.4	6.25	24.4	6.39	13.7
2	5.48	15.4	7.02	23.5	6.81	15.7
3	6.10	16.8	7.59	22.2	7.23	16.2
4	6.68	19.6	8.01	23.0	7.60	17.6
5	7.25	21.7	8.35	21.6	7.94	15.6
6	7.76	19.9	8.60	20.6	8.19	14.0
7.	8.13	12.5	8.79	17.6	8.38	12.6
8	8.31	7.4	8.92	16.4	8.53	6.7
9	8.41	3.2	9.02	15.7	8.60	2.1
10	8.45	.8	9.10	13.6	8.62	1.0

Plate No.	Run No.22		Run No.23		Run No.24	
	C_w x10-4	E	C_w x10-4	E	C_w x10-4	E
1	5.82	14.9	6.07	13.1	6.40	22.4
2	6.33	15.4	6.51	12.3	7.08	23.6
3	6.78	17.6	6.87	13.9	7.67	22.8
4	7.22	20.2	7.23	14.3	8.11	25.2
5	7.64	20.4	7.55	13.5	8.49	20.0
6	7.98	16.3	7.81	14.8	8.72	11.7
7	8.20	16.7	8.06	15.2	8.83	10.8
8	8.39	6.3	8.28	13.7	8.92	5.3
9	8.45	2.2	8.45	12.2	8.96	5.6
10	8.47	1.1	8.58	4.2	8.98	2.9

Table 5. Continued.

Plate No.	Run No.25		Run No.26	
	C_w x10-4	E	C_w x10-4	E
1	7.25	32.9	5.15	26.1
2	8.02	32.2	6.34	26.5
3	8.54	32.4	7.25	26.8
4	8.90	19.5	7.94	26.7
5	9.05	20.6	8.45	17.6
6	9.18	10.0	8.70	13.4
7	9.23	8.7	8.86	7.7
8	9.29	2.4	8.94	4.2
9	9.30	2.5	8.98	1.1
10	9.31	2.5	8.99	1.1

Table 6. Data on Calibration of Rotameter.

Rotameter rdg.	Time sec.	Volume cc.	Flow rate cc/min.
230	206.5	2000	581.0
200	237.5	2000	506.0
150	340.0	2010	354.5
100	541.5	2000	221.5
70	212.0	500	141.5
60	253.0	500	118.5
50	595.0	1000	100.5
25	866.0	500	34.6

Table 7. Equilibrium Distribution Data of Benzoic Acid-Toluene-Water System

C_T, C_w = lb.mol./ft.^{3}

74°F		79°F		81°F	
C_T	C_w	C_T	C_w	C_T	C_w
.00824	.000801	.01474	.001084	.01445	.001085
.00753	.000762	.00966	.0008825	.01045	.000920
.00669	.000717	.00894	.0008415	.00917	.000870
.00588	.000685	.00804	.000814	.00835	.000834
.00339	.00053	---	---	.00705	.000772
		.00707	.0007725	.00448	.000838
		.00538	.000662	.00362	.000582
		.004485	.0006205	.00175	.000423
				.000905	.000312
		.00362	.000565		
		---	---		
		.00180	.000414		
		.000828	.000290		

84°F		88°F		91°F	
C_T	C_w	C_T	C_w	C_t	C_w
.01449	.001099	.0149	.001145	.0152	.00119
.01185	.001006	.0108	.000980	.0101	.000980
.00930	.000883	.0095	.000918	.00830	.000896
.00586	.000718	.00852	.000870	.00728	.000828
.00479	.000656	.00662	.000795	.00641	.000800
.00298	.000528	.00586	.000745	.00545	.000731
.00178	.000417	.00473	.000670	.00369	.000607
.001615	.000414	.00372	.000600	.00366	.000607
.000905	.000319	.00186	.000442	.00179	.000442
.000774	.000285	.00100	.000318	.000897	.000318